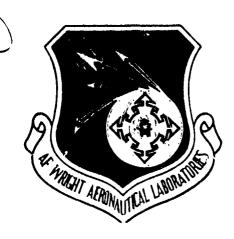
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ANALYSIS OF AIRCR. ELS AND RELATED MATERIALS

F. Neil Hodgson, Edward A. Steinmetz, John D. Tobias, Billy B. Bowles, and Charlotte D. Fritsch

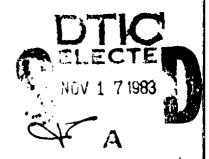
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SEPTEMBER 1982

FINAL REPORT FOR PERIOD 15 APRIL 1978—31 DECEMBER 1981

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AERO PROPULSION LABORATORY AIR FORCE WRIGHT AERONAUTICAL LABORATORIES AIR FORCE SYSTEMS COMMAND WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433



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Fuel tests, analyses, and analytical method development were conducted on a number of fuels of an experimental nature in conjunction with ongoing Air Force programs for studying fuel combustion behavior, turbine engine design, and other fuel related technologies. Fuels from conventional and alternate sources were studied, as were fuels of the high density missile propellant type. A wide variety of both physical and chemical properties of the fuels were measured and are tabulated. Studies conducted to aid in the solution of operational problems are also reported.

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FOREWORD

This final report was submitted by Monsanto Research Corporation under Contract F33615-78-C-2004. The effort was sponsored by the Air Force Aero Propulsion Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio under Project 3048, Task 304805, and Work Unit 30480593, with Dr. Ronald D. Butler as Contract Monitor through May 1979 and with Major Donald D. Potter as Contract Monitor from June 1979 through the conclusion of the efforts. Mr. F. Neil Hodgson of Monsanto Research Corporation was technically responsible for the work, which was performed during the period 15 April 1978 to 31 December 1981.

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Much of the work performed during the course of this program was planned in coordination with a number of other related fuel technology studies being conducted at, or under the sponsorship of, the Air Force Aero Propulsion Laboratory. Such efforts include research on turbine engine combustor design, fuel combustion behavior, improved fuel characterization methodology, high density missile fuel development, and alternate fossil fuel source development. The experimental results presented in this report are intended to be used in the context of those research programs, and it is expected that the significance of the data will become apparent as the technical aspects of the related programs are In particular, many of the fuel specimens examined during this program were experimental in nature, and the various chemical and physical properties tabulated herein should not necessarily be regarded as characteristic of particular fuel types.

The authors wish to gratefully acknowledge the excellent guidance provided by Major Donald D. Potter during the course of this work. Special gratitude is also expressed to members of the Monsanto Research Corporation staff: J. V. Pustinger for his technical consultation and advice, and D. Q. Douglas, M. K. Hershey, E. M. Hughes, L. Metcalfe, J. F. Moon, L. Parts, J. E. Strobel, and G. L. Thomas for their technical contributions. H. W. Luebke and O. P. Tanner, Monsanto Company, St. Louis, are also recognized for the special data they have provided.

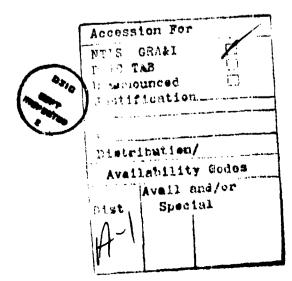


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SECTION I

INTRODUCTION AND SUMMARY

The Fuels and Lubrication Division of the Air Force Aero Propulsion Laboratory conducts in-house and contractual programs in all aspects of aircraft and missile fuels technology. These programs are designed to ensure an adequate supply of dependable fuels for operational use. The concerns encompassed in that goal cover every aspect of fuel technology: production, availability, properties, and quality; fuel storage, transportation, distribution, handling, and contamination; fuel additive effects and synthetic fuels for particular weapons systems; and combustion behavior, combustor design, and engine exhaust emissions. Work performed under this contract during the past four years has supported these efforts.

A large number of fuel specimens were physically and chemically characterized in connection with Air Force contractual programs to study the combustion properties of modified or experimental fuels. This work involved characterization of fuels after modification with aromatic stocks or a hydrocarbon stock consisting largely of relatively high boiling paraffins. Analyses and property measurements were continued throughout the course of the program to determine changes in the fuels resulting from the various combustion tests.

A number of investigations and analyses were conducted on high density fuels and fuel blends. The fuels included RJ-5 and RJ-5 blends, RJ-6, JP-9, and JP-10. Tests included IR analyses for identification of particulate contamination, GC analyses of degradation products, detailed measurements of a number of physical and chemical properties, and compositional analyses by gas chromatography.

Various special investigations were conducted. The effectiveness of two antistatic fuel additives in minimizing the buildup of static electrical charges in various JP-4 and JP-8 fuels was determined. The problem of polysulfide sealant chalking in F-16 aircraft fuel tank, was investigated in a special study to define the effects trace metals, mercaptan sulfur, and peroxide content have on the process. The Microsep II Water Separometer was evaluated against the Minisonic system for measuring the ease with which fuels release entrained or emulsified water. specimens were analyzed to determine if they contained hydrazinetype propellants. Various JP-4 type fuels were analyzed for metals content to determine the amount of dissolved metal that had been transferred from water lying in the bottom of the fuel tanks. A series of chemical analyses were conducted for the main engine filters to determine the cause of plugging in certain aircraft. The ball-on-cylinder fuel lubricity tester at AFWAL/ POSF was modified and evaluated for significance of test data. Troublesome sulfur compounds present in shale derived JP-4 fuels were identified by gas chromatography/mass spectrometry (GC/MS) after application of unique concentration techniques.

These and other studies required for research support or problem solving are described in the sections that follow.

SECTION II

SPECIAL INVESTIGATIONS

A number of special investigations were conducted either to provide solutions for operational problems, to improve existing test methodology, to respond to particular unanticipated needs as they arose, or to make measurements of a nonroutine nature. These tasks encompassed a wide range of activities including fabrication and modification of test apparatus, identification of fuel contaminant and filter deposits, studies of sealant and elastomer degradation, and a variety of other specialized investigations.

1. CHARGING TENDENCY OF FUELS CONTAINING ANTISTATIC ADDITIVES

Static electrical charges can be generated when fuel is added to aircraft fuel tanks containing reticulated polyurethane foam. several occasions electrical discharges are known to have caused low-grade explosions during aircraft refueling. This problem can be minimized by the use of an antistatic additive in the fuel. The purpose of this investigation was to generate data to compare the effectiveness of two such antistatic fuel additives, Stadis® 450 (Du Pont Petroleum Products) and ASA-3 (Shell). Comparisons were conducted using two different fuel tank foams, specifically the blue polyether urethane and the red polyester urethane. were conducted at room temperature (70°F) and in a cold chamber The concentration of additive in the fuel was prepared to bracket an electrical conductivity value of 200 picosiemens per meter (pS/m) at 70°F, or 100 pS/m at 20°F. A variation of the Exxon ministatic tester (MST) was constructed for these measurements. The fuels used in this investigation were selected to include a variety of fuel types. Two of the fuels, a JP-4 from Tinker AFB and a JP-4 from Richards Gebaur AFB, had exhibited electrostatic charge problems in actual usage. This subsection describes the apparatus, procedure and results.

Apparatus

The Exxon ministatic tester consists essentially of a cell to hold the test foam specimen and a syringe drive to force fuel through the foam specimen. A schematic diagram of the modified test unit constructed for these studies is shown in Figure 1. The unit has the essential features of the Exxon MS tester; however, rather than using a syringe drive to pass fuel through the test cell, a Teflon gear pump was employed for that purpose. The entire unit was constructed in an upright freezer cabinet so that tests could be conducted at reduced temperatures, as well as at room temperature.

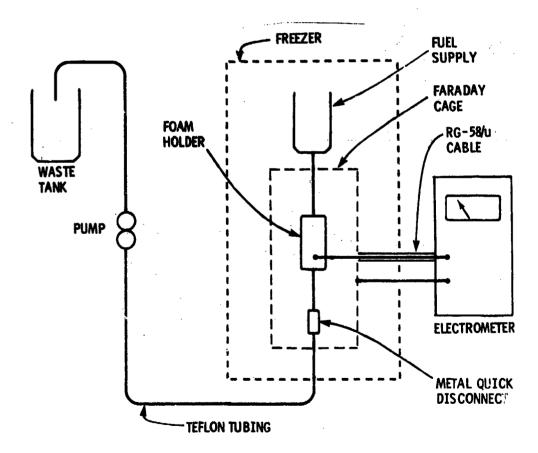


Figure 1. Schematic of test apparatus for fuel charge measurements.

Originally the test cell was constructed to accommodate a foam specimen 3 inches in length and 1 inch in diameter. The cell size was later reduced so that specimens 3 inches x 1/2 inch diameter could be used.

A foam specimen cutter was designed and constructed as a piece of auxiliary equipment for these tests. The device employed an electrically heated wire to cut either 1/2 inch or 1 inch diameter specimens. Rectangular blocks of foam are mounted on a rotating mandrel. The hot wire, which is mounted parallel to the axis of rotation of the foam, can be adjusted to give a cylindrical foam specimen of the desired diameter. This technique for cutting foam specimens was selected because of the smooth cut given by the hot wire and the resulting excellent reproducibility of specimen dimensions.

The principal objective in designing the apparatus was to produce a tester in which as many parameters as possible were controlled. The test cell, shown in Figure 2 in its 1 inch diameter configuration, was constructed of stainless steel. The cell position, relative to the other components in the freezer cabinet, is shown in Figure 3. To prevent formation of a flammable fuel vapor/air mixture in the freezer enclosure, the unit was continuously flushed with a slow flow of nitrogen.

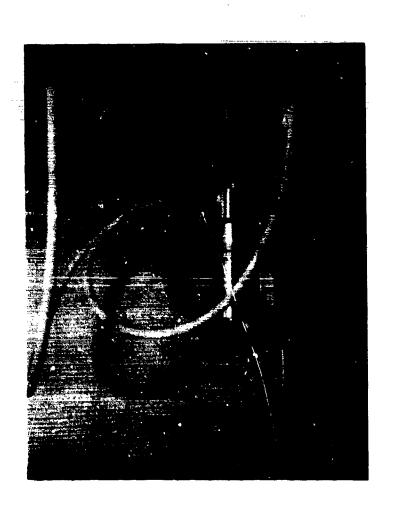
A Teflon gear pump is located on the left outside wall of the freezer and is shown at close range in Figure 4. All connections are made with polytetrafluoroethylene tubing. The pumping rate as a function of pump voltage was recorded. The voltage required to produce the desired flow of exactly 400 ml per minute through the device was determined. A controlled power supply to accurately produce the required voltage was employed to drive the pump.



Figure 2. Cell designed to hold foam test specimen for static charge measurement.



Figure 3. Freezer containing static charge test apparatus.



Teflon@ gear pump used in static charge test apparatus. Figure 4.

A Keithly Model 600B electrometer was used for the charge measurements as well as for preliminary electrical conductivity measurements.

Procedure

ASTM test method D 3114-72, "DC Electrical Conductivity of Hydrocarbon Fuels," was used to measure electrical conductivities of the test fuels after addition of the antistatic additives. In order to correlate additive concentrations with fuel conductivity, blends having various additive levels were prepared. By plotting additive concentration versus conductivity, the additive levels corresponding to conductivities of 200 pS/m at 70°F and 100 pS/m at 20°F were determined for each fuel. These additive concentrations, which were used for the evaluations, are presented in Table 1. The conductivity test apparatus, consisting of a Keithly Model 600B electrometer and conductivity cell, is shown in Figure 5. Conductivity-additive concentration plots for some of the fuels included in this study are presented in Figures 6 through 14.

TABLE 1. ADDITIVE CONCENTRATIONS REQUIRED TO OBTAIN FUEL ELECTRICAL CONDUCTIVITY VALUES OF 200 ps/m AT 70°F AND 100 ps/m AT 20°F

,	Concentration, ppm				
		5A-3	S.	S-450	
	70°F	20°F	70°F	20°F	
Fuel	(22°C)	(-6.7°C)	(22°C)	<u>(-6.7°C)</u>	
JP-4, AFFB 13-69	0.5	0.4	0.9	0.5	
JP-8	1.0	0.9	1.2	0.7	
JP-4, AFFB 14-70	1.0	0.7	1.0	0.7	
Clay Treated JP-4	0.5	0.5	0.4	0.3	
JP-4, Tinker AFB	0.6	0.4	0.5	0.4	
JP-4, Richards Gebaur AFB	0.5	0.4	0.5	0.4	

^aPicosiemens per meter



Figure 5. Fuel electrical conductivity test apparatus.

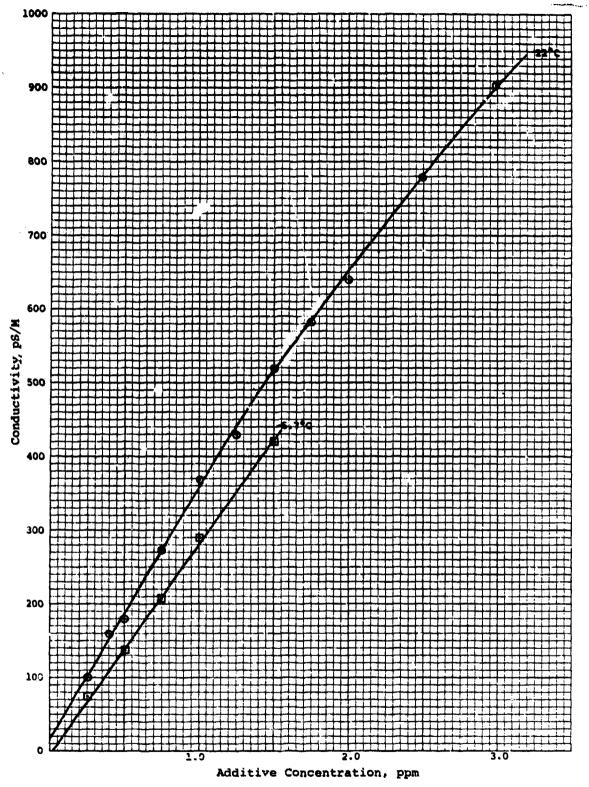


Figure 6. Fuel conductivity/additive concentration plots for fuel AFFB 13-69 with ASA-3 additive.

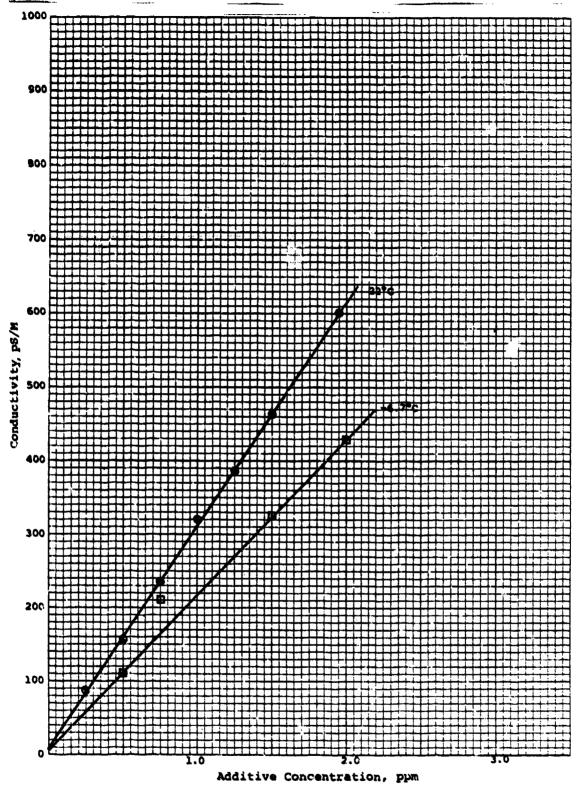


Figure 7. Fuel conductivity/additive concentration plots for fuel AFFB 13-69 with S-450 additive.

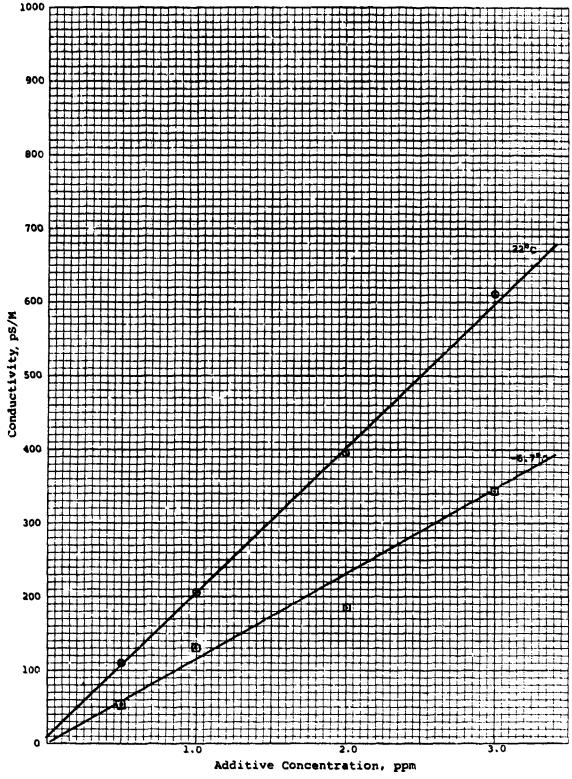


Figure 8. Fuel conductivity/additive concentration plots for JP-8 with ASA-3 additive.

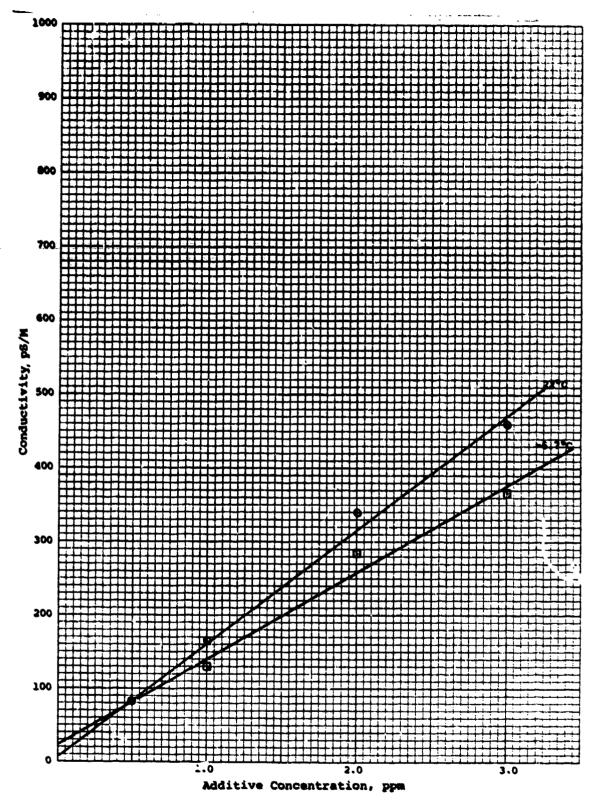


Figure 9. Fuel conductivity/additive concentration plots for JP-8 with S-450 additive.

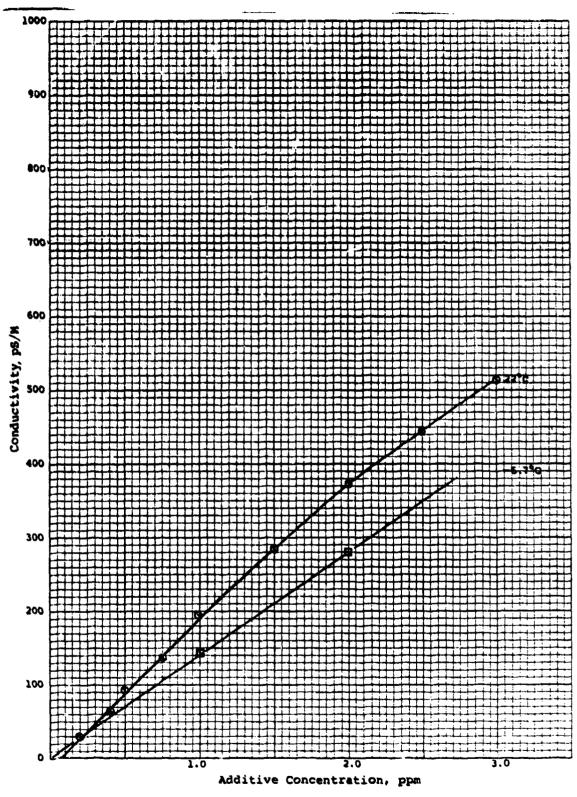


Figure 10. Fuel conductivity/additive concentration plots for fuel AFFB 14-70 with ASA-3 additive.

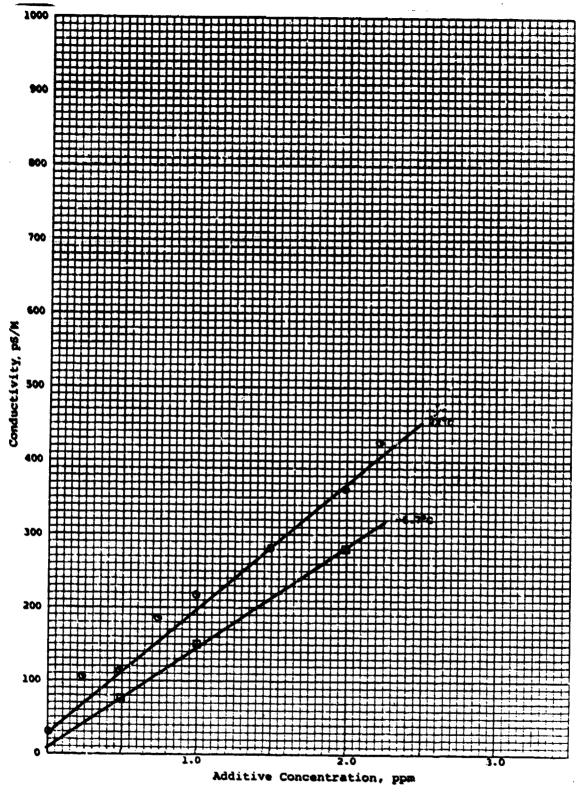
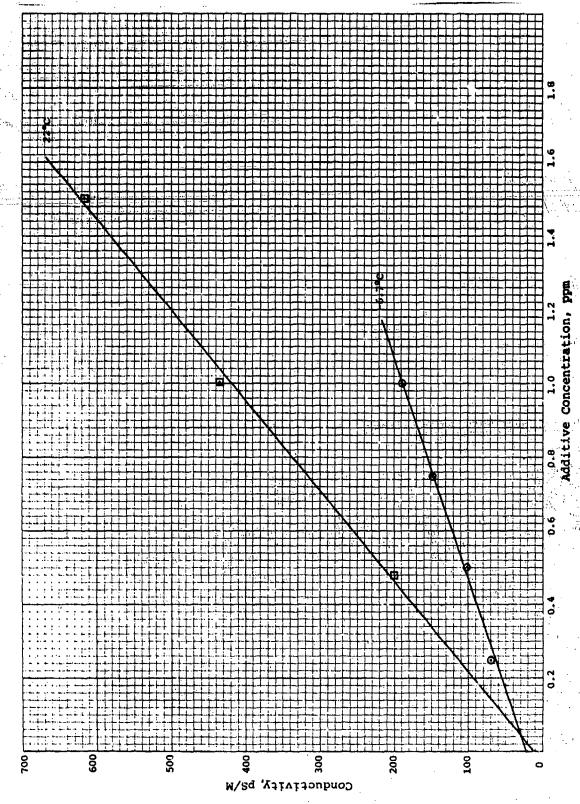
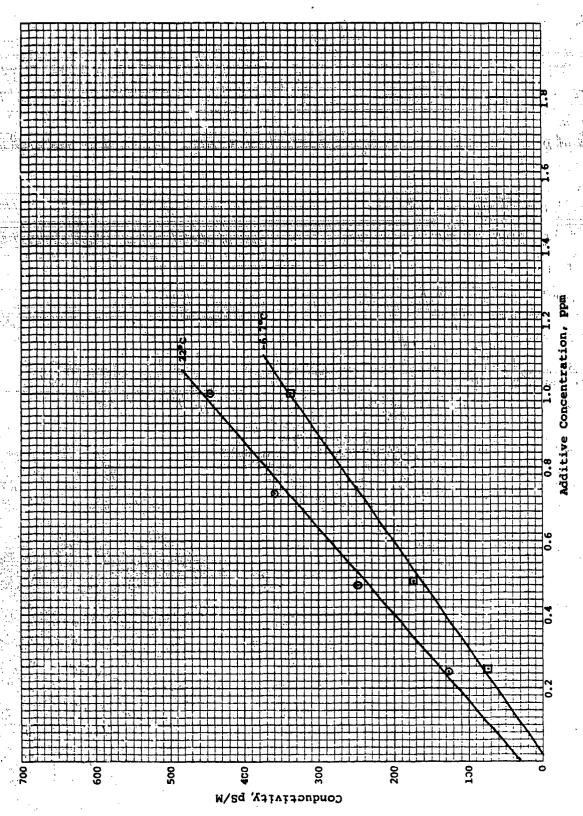


Figure 11. Fuel conductivity/additive concentration plots for fuel AFFB 14-70 with S-450 additive.



Fuel conductivity/additive concentration plots for clay treated JP-4 with ASA-3 additive.



Fuel conductivity/additive concentration plots for clay treated JP-4 with S-450 additive.

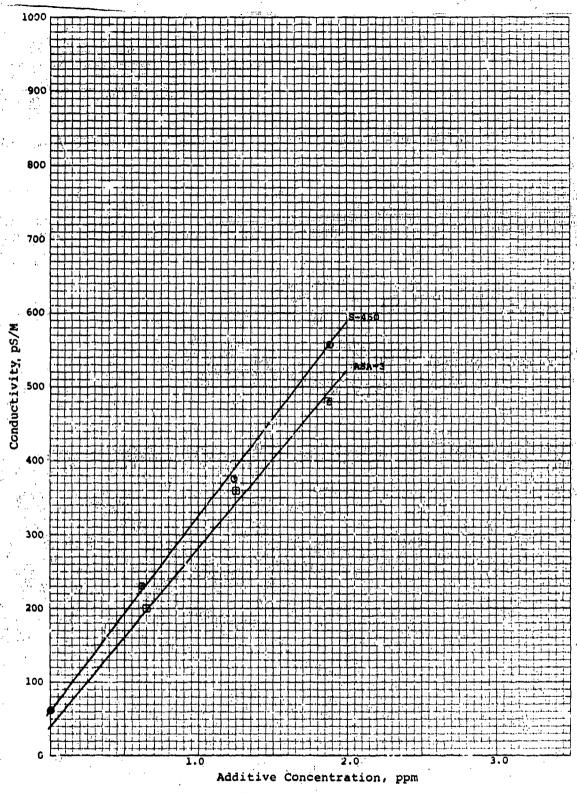


Figure 14. Fuel conductivity/additive concentration plots for Tinker AFB JP-4 with ASA-3 and S-450 additives.

Charging tendency measurements were conducted for each fuel at 70°F and 20°F, using both red and blue fuel tank foams. Fuel flow was exactly 400 ml/min, and a new foam specimen was employed for each test. To facilitate evaluation of the effect of the additives, measurements were conducted over a range of additive concentrations. For many fuel/foam/additive combinations, the sign of the accumulated charge changed as the concentration of additive was increased. Comparable tests were conducted with each additive and these were completed within the same test period. This precaution was felt to be important so that uncontrolled factors such as relative humidity or foam storage conditions would not bias the evaluation.

In order to determine the influence of corrosion inhibitor on charging tendency, two different corrosion inhibitors were added to clay-treated JP-4 at the concentration specified for that additive. Unicor J and DCI-4a were added at the level of 15.2 ppm in the fuel. Using this fuel, charging tendency measurements were conducted at 70°F with both antistatic additives and with both foams.

Results and Discussion

Results of the tests are presented in Tables 2 through 7 and are plotted in Figures 15-24. Values are given in microcoulombs per cubic meter of fuel. It should be noted that data for the first three fuels, presented in Tables 2-4, were obtained under slightly different conditions than for subsequent fuels. It was discovered that previous similar tests (ref. 1) had been conducted using foam specimens having a 0.5 inch diameter rather than a 1 inch diameter. To make the current tests comparable to those in the referenced report, either the foam specimen volume needed to be decreased or the fuel flow scaled up proportionately. The first option was selected, that of modifying the sample cell to accept a 0.5 inch foam sample.

TABLE 2. CHARGING TENDENCY OF JP-8 ON POLYURETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

	Additive	Charge, µC/m ³				
		70°F		20°F		
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3	
Red polyester	0	+12	+11	+27	+24	
pougaste	0.5	-11	- 7	-22	-13.5	
	1.0	<u>-</u> 7	-3	-16	-7.5	
	1.5	- 5	-1.5	-13	-7.5	
	2.0	-3	-0.5	-8	-3.5	
	2.5	-1.5	-	-	-	
Blue polyether	0	+46	+44	+57	+55	
	0.5	-28	-15	-38	-21	
	1	-13.5	-8	-27	- 15	
	1.5	-3	- 3	-21	-8	
	2	-1.5	- 1.5	~ 15	- 7	
	2.5	-	-	- 7	-4	

TABLE 3. CHARGING TENDENCY OF FUEL AFFB 13-69 ON POLYURETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

	•	Charge, µC/m ³				
	Additive conc., ppm	70		20°	F	
Foam type		S-450	ASA-3	S-450	ASA-3	
Red foam	0	-29	-28	-31	-32	
	0.5	-22	-4	-16	-13	
	1.0	-12	-2.5	-8	-6	
	1.5	-3	-2.5	- 5	-3	
•	2.0	-0.8	-0.8	-3	-0.7	
Blue foam	0	-36	-36	-4 5	-46	
	0.5	-8	-7	-29	-11.5	
	1.0	-2	- 5	-13.5	 6	
	1.5	-0.8	-2	-6	· - 2	
	2.0	-0.8	-0.8	-4.5	-0.7	
•	2.5	446		-1.5	-	

TABLE 4. CHARGING TENDENCY OF FUEL AFFB 14-70 ON POLYURETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

		Charge, µC/m ³			
	Additive	70°F		20	F
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3
Red foam	0	+11	+11	+32	+32
	0.5	-8	-3	-15	-2.3
	1.0	- 7	-1.2	-12	-1.1
	1.5	-5	-1.1	-7	-1.1
	2.0	-3	-0.8	-4.5 -3.8	-0.5
Blue foam	0	+50	+50	+68	+68
	0.5	-13	-4. 5	-17	-4.5
	1.0	-8	-1.8	-8.5	-1.5
	1.5	- 5	-1.3	-6	-1.4
	2.0	-3	-0.8	-4	-1.1
	2.5	-2	-	-3	-0.8

TABLE 5. CHARGING TENDENCY OF JP-4 (Richards Gebaur AFB)
ON POLYURETHANE FOAMS WITH ASA-3 AND S-450
FUEL ADDITIVES

		Charge, μC/m ³			
	Additive	70	70°F		F
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3
Red foam	0	+39	+39	+44	+43
	0.15	_	- 9	••	-6
	0.25	-11	-4.5	~7.5	-4
	0.50	-8	-3	-2.2	-4
	0.75	-1.5	- 3	- 1.5	-4
	1.0	-1.5	- 3	-0.7	-3.5
Blue foam	0	+30	+33	+33	+33
	0.25	-18	-18	-9.0	-6.7
	0.50	-10.5	-11	- 7.5	-2.2
	0.75	-1.8	-1.8	-2.9	-1.8
	1.0	-0.9	-0.9	-1.6	-0.9

TABLE 6. CHARGING TENDENCY OF JP-4 (Tinker AFB) ON POLYURETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

			, μC/m ³	IC/m ³		
	Additive	70	70°F		20°F	
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3	
Red foam	0	+35	+35	+40	+42	
	0.15	+15	+9.0	-	+13.5	
	0.25	+11	+4.5	+6.7	+6	
•	0.50	+7	+3.0	+5	+4.5	
	0.75	+1.5	+3.0	+2	+3.8	
	1.0	+1.3	+3.0	+2	+3.8	
Blue foam	0	+30	30	37	38	
	0.15	18	_	_	-	
	0.25	11	4.5	9	7.5	
	0.50	1.8	3.0	6.8		
	0.75	0.9	1.5	4	5 3	
	1.0	_	-	0.7	0.9	

TABLE 7. CHARGING TENDENCY OF CLAY TREATED JP-4
ON POLYURETHANE FOAMS WITH ANTISTATIC
ADDITIVE AND CORROSION INHIBITORS

Foam type	Additive conc., ppm	Charge, (µC Unicor J 15.2 ppm S-450 ASA-3		/m ²) at 70°F DCI-4A 15.2 ppm S-450 ASA-3	
Red polyester	0	+45	4 5	4 5	44
	0.17	33	12.5	8.5	14
	0.34	8.1	11	5.5	7
	0.50	4.2	10	2.8	5
	0.67	2.2	4.8	2.0	3
	1.0	0.7	1.1	0.8	0.9
Blue polyether	0	+58	58	60	60
2 2	0.17	40	33	42	45
	0.34	31	29	29	33
	0.50	9	12	15	10
	0.67	5	7	4	6
	1.0	3	4	2	3

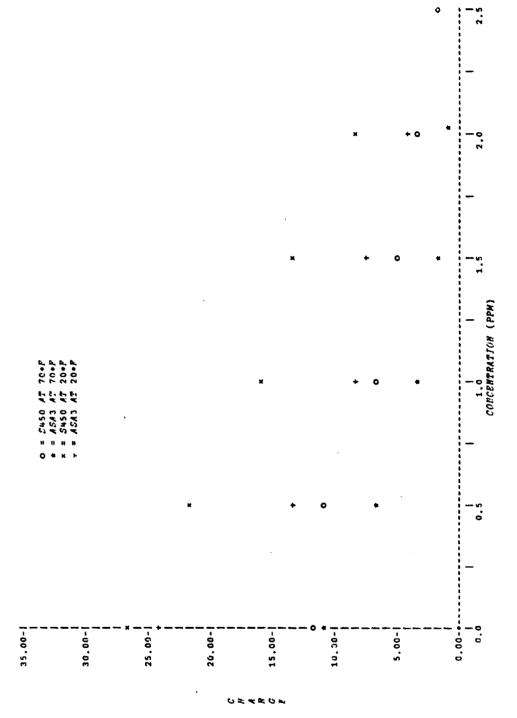


Figure 15. Accumulated charge $(\mu C/m^3)$ versus additive concentration for JP-8 fuel with red foam.

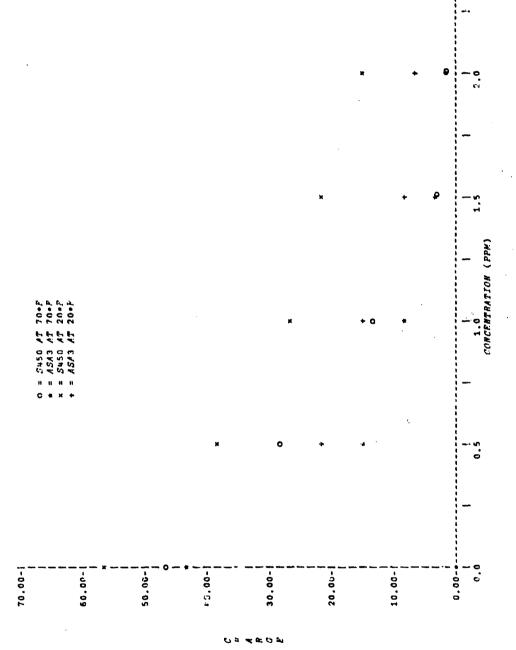
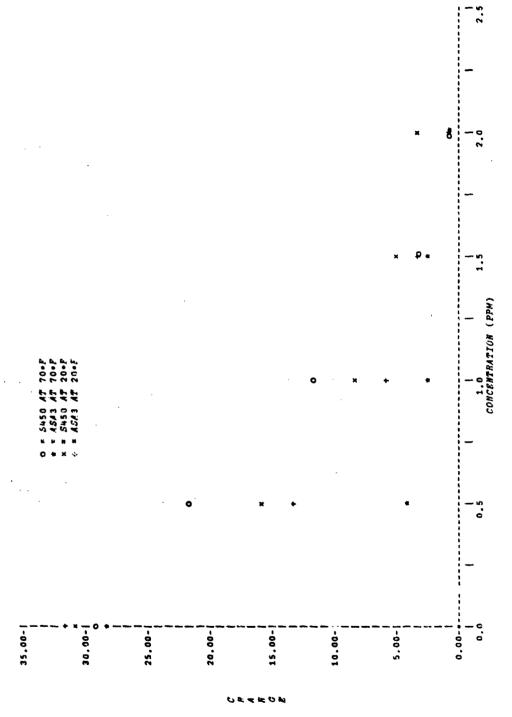
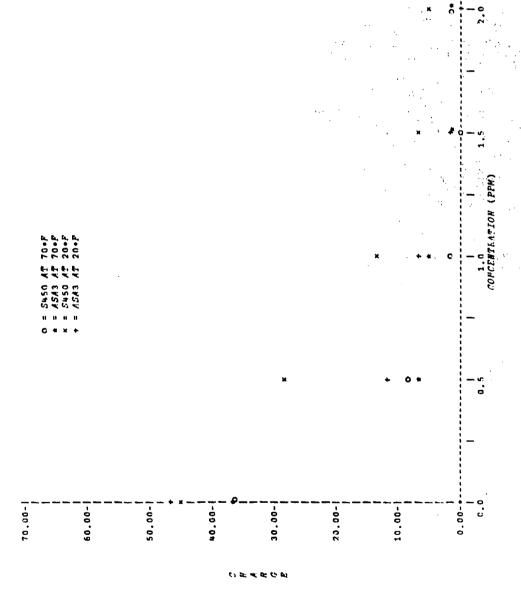


Figure 16. Accumulated charge $(\mu C/m^3)$ versus additive concentration for JP-8 fuel with blue foam.



Accumulated charge $(\mu C/m^3)$ versus additive concentration for AFFB 13-69 fuel with red foam. Figure 17.



Accumulated charge $(\mu C/m^3)$ versus additive concentration for AFFB 13-69 fuel with blue foam. Figure 18.

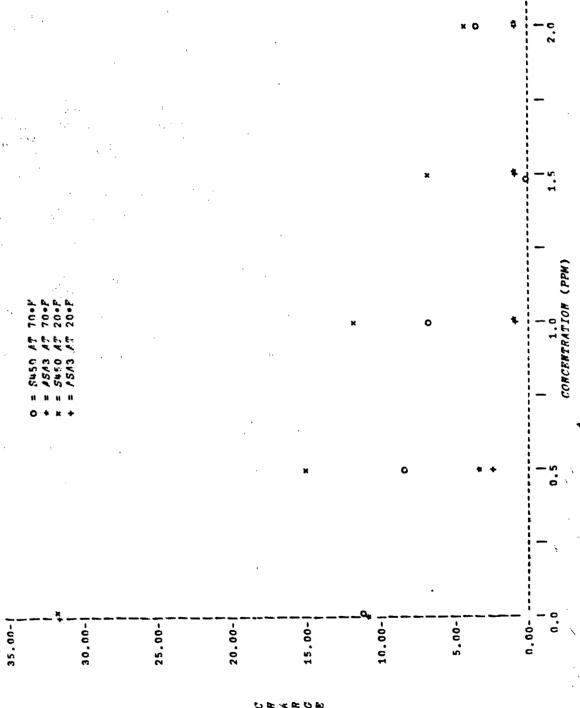
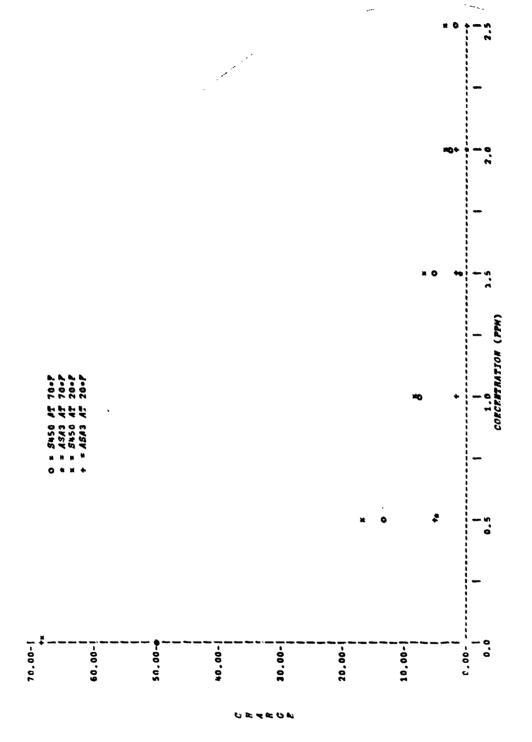


Figure 199 Accumulated charge $(\mu C/m^3)$ versus additive concentration for AFFB 14-70 fuel with red foam.



Accumulated charge $(\mu C/m^3)$ versus additive concentration for AFFB 14-70 fuel with blue foam. Figure 20.

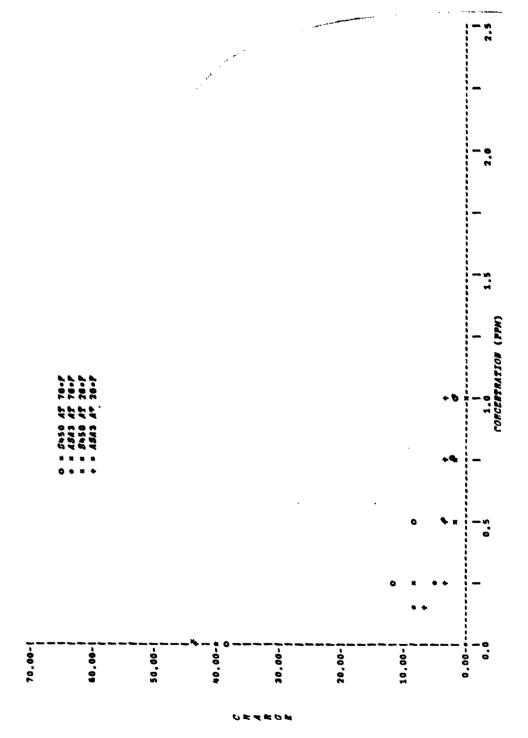
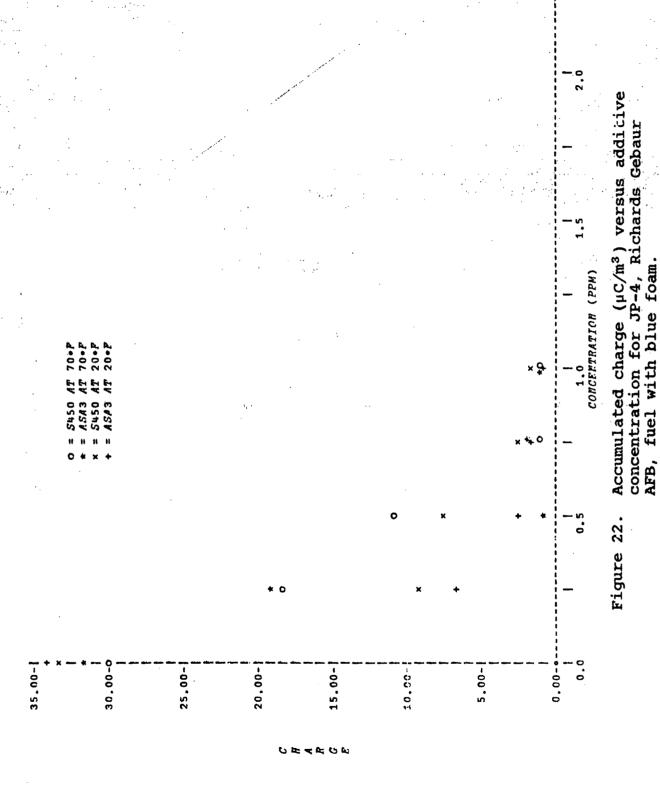
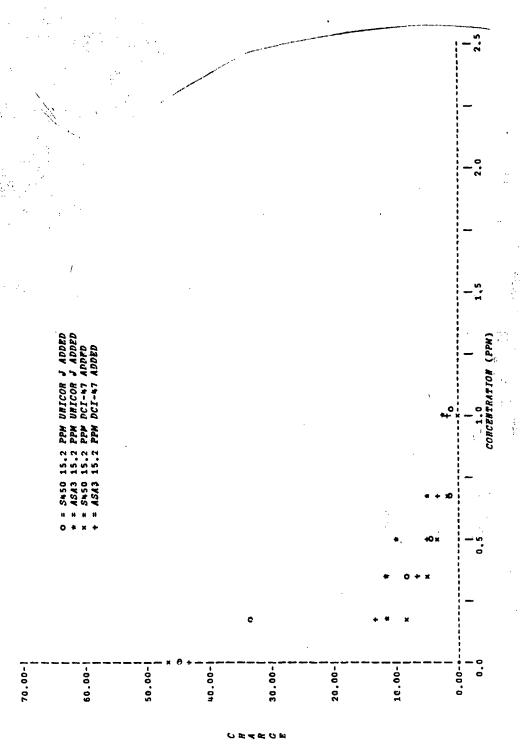
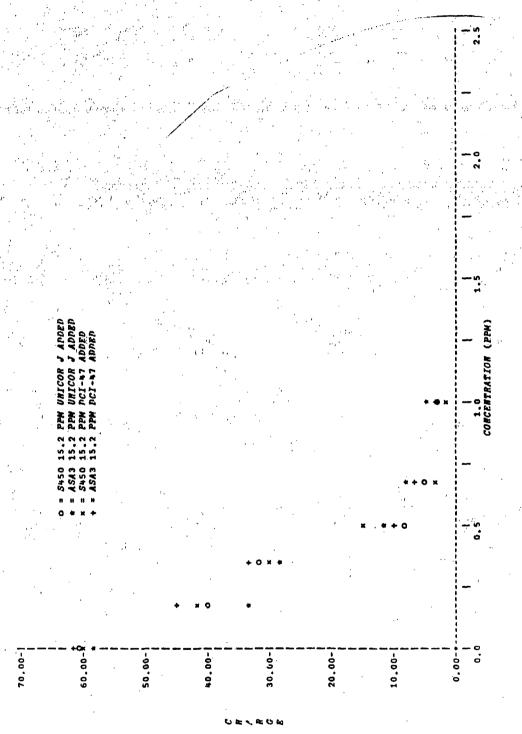


Figure 21. Accumulated charge $(\mu C/m^2)$ versus additive concentration for JP-4, Richards Gebaur AFB, fuel with red foam.





Accumulated charge $(\mu C/m^3)$ versus additive concentration for clay treated JP-4 fuel at $70^{\circ}F$ with red foam. Figure 23.



Accumulated charge ($\mu C/m^3$) versus additive concentration for clay treated JP-4 fuel at 70°F with blue foam. Figure 24.

Although the apparatus for measurement of static charging tendency was similar in all ways to the Exxon MS tester, data generally differed from that reported from the MS tester in one aspect. Samples in current tests gave a high charge value when no antistatic additive had been added. Charge generally decreased with additive concentration. MS tester data, however, have shown low charge values for the baseline fuel and higher values with the addition of antistatic additives. This discrepancy was never completely resolved. Data presented in this report, however, are felt to reflect the behavior of the additive, despite this difference from previous work.

In sampling the foam materials, an effort was made to obtain identical specimens for the comparative tests. Fuel flows were passed through the foam in the same direction relative to the specimen's position in the original foam block. Specimens were cut just before the test so that comparative specimens experienced the same sample history. Under these conditions, results were quite reproducible. In the case of the Tinker Air Force Base JP-4, twelve measurements were taken on the starting fuel for each foam and twelve were taken on the fuel having additive at the 200 pS/m conductivity level for each foam. In this series of measurements, foam specimens were not specifically selected on the basis of orientation in the original block, but rather were completely randomly taken from the block. Under these latter conditions, relative standard deviations of 16.2% for red foam and 16.1% for blue foam were obtained in the measurements with no antistatic additive. Relative standard deviations were recorded with ASA-3 antistatic additive at a level to give a 200 pS/m conductivity, these being 17.7% for blue foam and 24.5% for red foam.

Conclusions

While the matrix fuel had a great effect on the absolute value of the charge, no great difference between the two additives was apparent for most fuels. In the case of the Tinker AFB JP-4, S-450 showed slightly better performance than ASA-3 at 70°F for both foams. However, at 20°F ASA-3 out-performed S-450 for the red foam but not for the blue foam.

2. CHARGING TENDENCY OF A JP-4 SAMPLE CODED 81-3-CRM

About two years after the study described in subsection 1 above, electrical conductivity and charging tendency were evaluated for an operational JP-4 (coded 81-3-CRM) and for a reference fuel (AFFB 14-70). The measurements were conducted on the fuels before treatment and after the addition of the antistatic additives Stadis 450 (Du Pont Petroleum Products) and ASA-3 (Shell Corp.) to the fuels at various concentrations. Two different fuel tank foams, blue polyether urethane and red polyester urethane, were used in the study. The polyurethane foam specimens were cut into 1/2 inch diameter x 3 inch cylinders to provide a snug fit within the same cell of the test device. The tests were conducted at 70°F and 20°F in exactly the same manner as described in subsection 1.

Figures 25 and 26 illustrate the additive levels corresponding to various conductivity values, expressed in picosiemans per meter. Charging tendency was similarly measured over a range of additive concentrations, with the values being expressed in microcoulombs per cubic meter. Data are presented in Tables 8 and 9 for the various fuel/fcam combinations at both 70°F and 20°F. It will be noted that the sign of the accumulated charge changed as the additive concentration was increased.

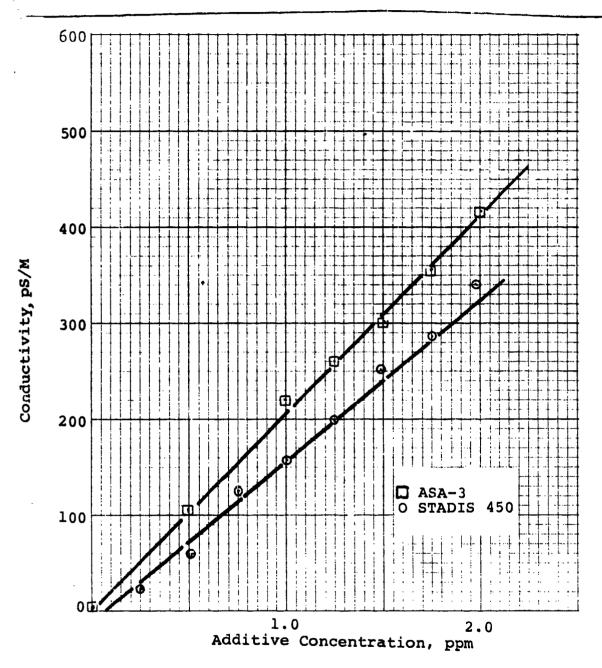


Figure 25. Fuel electrical conductivity as a function of additive concentration for reference fuel AFFB 14-70.

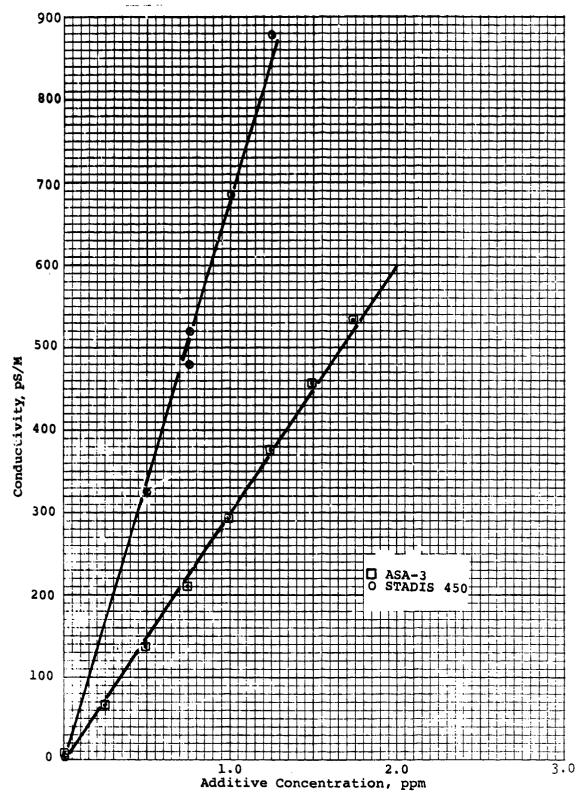


Figure 26. Fuel electrical conductivity as a function of additive concentration for fuel coded 81-3-CRM.

TABLE 8. CHARGING TENDENCY OF REFERENCE FUEL AFFB 14-70 ON POLYURETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

		Charge, µC/m ³						
	Additive	70	F	20	r'			
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3			
— 3 /			• •					
Red foam	0	17	18	25	24			
	0.5	-7	-3	-15	-3			
÷	1.0	- 5	-2.3	-9	-1.3			
	1.5	-3	-1.8	-6	-1.1			
	2.0	-1.5	-0.9					
Blue foam	0	+42	+44	+62	+60			
	0.5	-14	-9	-18	-12			
•	1.0	-7	-6	-12	- 5			
	1.5	-2	-0.8	-6	-2			
	2.0	-2	-0.8	-4	-0.8			

TABLE 9. CHARGING TENDENCY OF JP-4 CODE: 81-3-CRM ON POLY-URETHANE FOAMS WITH ASA-3 AND S-450 FUEL ADDITIVES

		Charge, µC/m ³							
	Additive	70		20	20°F				
Foam type	conc., ppm	S-450	ASA-3	S-450	ASA-3				
Red foam	0	+18	+18	24	23				
	0.5	-9	+9	-15	13				
	1.0	- 5	-4	-4	- 5				
	1.5	-4	-4	-4	- 5				
	2.0	-4	-3	-3	-4				
Blue foam	0	+78	81	95	97				
	0.5	-20	24	-35	37				
	1.0	- 5	7	-11	-13				
	1.5	- 5	- 5	-8	-9				
	2.0	-4	-4	- 5	- 5				

Discussion of Results

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Fuel AFFB 14-70 was included in this evaluation as a reference sample since it was a part of the earlier investigation reported in subsection 1. Data for this fuel, shown in Figure 26 and Table 8 agreed quite well with that obtained previously.

The electrical conductivity plot of fuel 81-3-CRM indicates that the addition of Stadis 450 affected the conductivity of that fuel more than the addition of the same amount of ASA-3. The charging tendency of fuel 81-3-CRM, as shown in Table 9, was much nigher for the blue polyurethane foam than it was for the red foam, and was higher than that of the reference fuel with blue foam. With the red foam, however, there appeared to be no substantial difference in the charging characteristics of the two fuels. The same trends observed at 70°F were also present at 20°F, except that the charging endency was somewhat enhanced at the lower temperature.

3. POLYSULFIDE SEALANT CHALKING, INITIAL STUDY

The problem of polysulfide sealant "chalking" in F-16 aircraft fuel tanks was investigated in a special study to define the effect trace metals and mercaptan sulfur have on the process. The chalking phenomenon had been identified in previous Air Force studies as being due to exposure of the calcium carbonate filler material, presumably as a result of a process in which the sealant is rendered fuel soluble. A study was conducted to determine the extent of the chalking problem and to evaluate the effect of metal deactivators on the rate of sealant chalking.

Determination of Fuel/Metal Equilibrium Concentrations

The chalking phenomenon was known to be influenced by the type and amount of dissolved metals present in the fuel. Certain metals such as copper and cadmium are reported to induce severe chalking. A study was therefore undertaken to determine the equilibrium concentrations of various metals in jet fuels upon contact with cleaned and activated metal strips at 140°F.

Test Fuels

Jet Reference Fuel (JRF) and JP-4 were used for the tests. The JP-4 that was provided for the study was found to contain approximately 400 ppb of lead. Additional data on the JP-4 was provided as follows:

Acidity, total mg KOH/gram	0.005
Corrosion	negative (1-A)
Mercaptan sulfur, wt %	0.0000
Total sulfur, wt %	0.01

The JRF fuel was prepared in glass vessels with volumetric quantities of the following materials:

cyclohexane	60	parts
toluene	30	parts
isooctane	. 10	parts
t-dibutyldisulfide	. 1	part
t-butylmercaptan	0.015	part

Test Metals and Cleaning Processes

Equilibrium studies were conducted with 3 x 3 inch coupons of copper, monel 400, cadmium, lead, mild steel, and anodized aluminum. The thickness varied slightly depending on the type of stock used. Each metal coupon was sequentially cleaned with the following solvents:

50/50 by volume methylene chloride and trichloroethylene trichloroethylene 50/50 by volume ethanol and acetone 30/30/40 by volume ethanol, acetone and deionized water deionized water

^aMeasurements performed at Det. 13 SA-ALC/SFQLA Laboratory, Wright-Patterson AFB, Ohio.

The cleaning operations were performed by rubbing the coupons with oil-free paper saturated with solvent. After all oil, grease and dirt had been removed, the coupons were immersed in deionized water and then wiped dry. Various pickling processes recommended by either the American Society for Metals or the Encyclopedia of Chemical Technology were used to activate the surfaces. processes varied from metal to metal and generally involved acid treatment of the metal as described in Table 10.

TABLE 10. CLEANING PROCESSES FOR CHALKING STUDY TEST METALS

Copper:

- 1.5 minute immersion in 40% ACS-grade HCl at 73°F
- rinse in deionized water at 73°F and blot dry

Monel 400:

- · 5-second dip in bath containing 1 gallon deionized water, 1 gallon reagent grade HNO3, and 1 1b NaCl at 73°F
- rinse in deionized water at 180°F
- immerse 6 minutes in 50 vol. % reagent grade HNO; at 73°F
- immerse l additional minute in fresh 50% HNO₃ at 73°F (optional)
- rinse in deionized water
- neutralize in 2% ammonia solution at 73°F
- · rinse in boiling deionized water and blot dry

Cadmium:

- 3-minute immersion in 35% ACS grade HCl at 73°F
 rinse in deionized water at 73°F and blot dry
- repeat sequential solvent cleaning using disposable
 - wipers and blot dry

Lead:

- 4-minute immersion in bath containing 8 vol. % reagent grade glacial acetic acid, 4.5 vol. % of reagent grade 30% hydrogen peroxide and 87.5 vol. % deionized water
- repeat sequential solvent cleaning using disposable wipers and blot dry
- · repeat above two steps (optional)

- Mild Steel: 5-minute immersion in 10 vol. % reagent grade sulfuric acid at 155°F
 - rinse in deionized water at 73°F
 - repeat sequential solvent cleaning using disposable wipers and blot dry
 - 1-minute immersion in above sulfuric acid bath at
 - rinse in deionized water at 73°F and blot dry
 - repeat sequential solvent cleaning as above and blot dry

Anodized

Aluminum: not pickled to prevent removal of anodized coating

Preparation of Glass Test Vessels and Sampling Containers

Wide-mouth quart jars with cork-backed Teflone-lined lids were used for conducting the equilibrium studies. One-ounce French Square bottles with Polyseal-lined caps were used for taking periodic fuel samples for analysis of metal content. A rigorous cleaning procedure was employed for these containers. The glass test vessels were cleaned with a scouring type of cleanser, then rinsed with tap water followed by deionized water. They were then rinsed with 50% nitric acid, rinsed again with copious amounts of deionized water, and then dried at 110°C in an oven. The small sample bottles and all cap liners were similarly cleaned to insure that they were metals-free.

Equilibrium Test Procedure

A single 3-inch square metal coupon and 850 milliliters of either JP-4 or JRF fuel were placed in the quart vessels. The tests involved six different metal coupons in each of two fuels, along with two control vessels with no metal strips, for a total of 14 test units. The vessels were placed in an explosion-proof oven at 140°F. Samples of 25 ml each were collected after periods of 2, 5, 7, 9, 12, 19, 26, 33, 40, 47, 54, 78, and 107 days. Each 25 ml aliquot of solution was analyzed by atomic absorption spectrophotometry for the coupon metal. Iron was analyzed for the mild steel test, and copper and nickel for the test with Monel. The control solutions were analyzed for all six metals at each intervals. A total of 202 solutions was analyzed for various metals.

Results and Discussion

The results of the equilibrium studies through 107 days are shown in Table 11. Examination of these results provides the following conclusions. Nickel, iron and aluminum, if solubilized at all, are

TABLE 11. METAL CONCENTRATIONS IN JET FUELS, PARTS PER BILLION

107	2	2	77	•	•		10,700	2	2	Ş	2	2	9	2	170	220	016	2	910	2	2	2	2	2	2	21
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35	8	2	74		*7	57	1,600	Ş	2	9	2	2	91	£	25	330	330	Q#	520	3	2	ğ	R	£	2	2 9
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9 12 19 26 13 40 47	2	2	ž) (2,200	9	ē	ğ	2	₽	•	9	•	400	430	9	420	9	30	30	\$	2	2	2 9
113	ě	Ş	ě	,	2 .	•	970	2	Ç	9	2	9	•	2	;	200	200		400	ē	20	30	9	<u>Q</u>	a	2 9
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nΙ	<u>.</u>	ğ	7	90	2 7	•	23,100	Q	Ä	Q	9	ē	2	2	7	310	324	9	Ä	9		Ä	9	9	2	29
71	NDA	Ä	7	70	7		9	QN	Ç	2	9			9		395	607	ON	읒	£	욡	Š	MD	QX		9
Strip	None	Mone1	Copper	du C		Mone	Copper 1	Mone	Monel	Mone	Monel	Mone	Cadmium	Mone	Cadmiu	Hone	Lead	Mone	Lead	Mone	Steel	Mone	Steel	None	Anod Al	Mone and at
Puel	JP-4			100	2			JP-4		JRF		JP-4		JRF .		JP-4C		JRP		JP-4		.1RP		JP-4		JRF
818	n Ca							Mi				8				Pb				Pe.				A1		

- not detected at 7 ppb for copper, 40 ppb for nickel, 3 ppb for cadmium, 30 ppb for lead, 20 ppb for iron and 400 ppb for aluminum Z Q N

- not detected but minimum detectability on these days was 20 ppb instead of 7 ppb JP-4C - submitted fuel was known to have a high lead content φX

present at concentrations below detection limits of the analytical technique being used (40, 20, and 400 ppb, respectively). The copper level in JP-4 with the copper coupon is relatively low, but it appears to be increasing at a slow rate. The copper level in JRF quickly rises to an extremely high value, apparently from reaction with the mercaptan which is present as part of the JRF formulation. Wide fluctuations in copper concentration in JRF observed as a function of time may be related to a precipitate which formed on the copper and then crumbled off into the fuel. Copper build-up in the fuel containing Monel appears to be insignificant. The lead content in JRF has continued to increase at a steady rate. After 54 days the lead content began to increase in JP-4 and at a much faster rate than in JRF. A similar pattern is followed with cadmium.

The results clearly show that different metals have a widely varying range of solubility in jet fuel, and that the presence of mercaptan significantly accelerates the rate of solubility from activated metal strips.

Effect of Metals, Mercaptan, and Metal Deactivator on Sealant Chalking in Fuel

Tests similar to those used for the metal solubility studies were conducted to evaluate the sealant chalking process in various environments. These environments include JP-4 and JRF fuel with presence/absence combinations of the following: an additional 150 ppm of butyl mercaptan (50 ppm mercaptan sulfur), 5.8 ppm N,N' bis-salicylal-1,2-propane diamine metal deactivator, and 3 in. x 3 in. metal coupons of copper, Monel 40, cadmium, lead, mild steel, or anodized aluminum. The JRF fuel has as a part of its normal formulation 150 ppm butyl mercaptan, thus it contained a total of 300 ppm butyl mercaptan (100 ppm mercaptan sulfur) when additional mercaptan was added for the chalking studies.

Test Procedure

A total of 47 test vessels and metal coupons were conditioned by the solvent cleaning and acid activation procedure previously described. A 2 in. x 1/4 in. x 1/8 in. strip of sealant was vertically suspended in each test vessel and immersed in the 500 ml of jet fuel it contained. Included in the fuels were various combinations of metal coupons, mercaptan and metal dea___vator. The test units were stored in a walk-in oven room maintained at 140°F and were checked daily for sealant chalking. Observations were made through the glass wall of the test vessels using strong fluorescent lighting. After 45 days, all sealant specimens were removed from the test jars and dried overnight for closer examination.

Results and Conclusions

Examinations made after the sealants had been dried showed that in most cases the chalking was more severe than was apparent when the sealants were observed through yellow-colored fuel. The times when chalking was first detected while looking through the fuels and the conditions of the dried strips are summarized in Tables 12 and 13. Photographs of the dried sealant strips are shown in Figures 27 through 33.

Generally, the presence of mercaptan caused an increase in the amount of chalking. While the presence of metal deactivator substantially decreased chalking in some tests, this was not true for all fuel/metal combinations. The overriding factor in sealant chalking was the specific fuels/metal combination. The significance of the factor is highlighted by the following observations:

(1) More chalking occurred in JP-4 than in JRF when comparing similar tests. The sealant chalking with JP-4 was more severe in the control test (neat fuel) and in tests with Monel,

RESULTS OF SEALANT CHALKING STUDY AFTER 45 DAYS AT 140°F IN JRF TABLE 12.

Final Appearance of Strips After Removal From Fuel and Overnight Drying	no change no change	thick yellow, crumbly coating thick yellow, crumbly coating thick yellow, crumbly coating thick yellow, crumbly coating		very thin line of chalking on edges light chalking, mainly on edges light chalking, mainly on edges light chalking, mainly on edges	thick brown, crumbly coating thick brown, crumbly coating thick brown, crumbly coating thick brown, crumbly coating	a)	no change no change no change no change
Time and Description of Initial Sealant Chalking**	none detected none detected	9 days - strip yellow 9 days - strip yellow 10 days - strip yellow 10 days - strip yellow	none detected none detected none detected none detected	none detected none detected none detected none detected	9 days - brown edges 9 days - strip brown 15 days - brown edges 9 days - brown edges	9 days - yellow edges 10 days - yellow edges 10 days - strip yellow 10 days - strip yellow	none detected none detected none detected none detected
5.8 ppm Metal Deactivator	ou ou	no Yes Yes	no yes yes	no yes yes	no no yes yes	no no yes yes	no no yes Yes
Additional 150 ppm Butyl- mercaptan*	no ves	no yes no yes	no yes no Yes	no yes yes	no yes no yes	yes no yes	no yes no yes
Metal	None None	Copper	Mone1	Cadmium	Lead	Mild Steel	Anodized Aluminum

JRF contains 150 ppm mercaptan as prepared. JRF with additional mercaptan thus contains 300 ppm total, or 103 ppm total mercaptan sulfur.

As observed through the glass test vessel and yellowish fuels, using fluorescent lighting. Only after 45 days were the sealant strips removed from solution for final observation.

RESULTS OF SEALANT CHALKING STUDY AFTER 45 DAYS AT 140°F IN JP-4 TABLE 13.

绝

Final Appearance of Strips After Removal From Fuel and Overnight Drying	light chalking, mainly on edges light chalking, mainly on edges	medium chalking, some cracks medium chalking, some cracks to apparent chalking very light chalking	medium chalking, some cracks medium chalking, some cracks light chalking, some cracks medium chalking, some cracks	light chalking, mainly on edges very light chalking	no change no change	heavy chalking with deep cracks heavy chalking with deep cracks medium chalking with no cracks	light chalking, mainly on edges medium chalking, mainly on edges very light chalking on edges medium chalking on edges
Time and Description of Initial Sealant Chalking*	none detected none detected	34 days - yellow edges 27 days - yellow sides none detected 27 days - yellow edges	36 days - yellow edges 27 days - yellow edges not detected 31 days - yellow edges	none detected none detected	none detected none detected	28 days - yellow edges 28 days - yellow edges 38 days - yellow edges	none detected none detected none detected none detected
5.8 ppm Metal Deactivator	ou ou	no yes yes	no yes yes	no Yes	no Yes	no no yes	no yes yes
Additional 150 ppm Butyl- mercaptan	no Yes	no yes no yes	no yes no yes	ou ou	ou ou	el no yes no	no yes no yes
Metal	None None	Copper	Mone1	Cadmium	Lead	Mild Steel	Anodized Aluminum

^{*} As observed through the glass test vessel and yellowish fuels, using fluorescent lighting. Only after 45 days were the sealant strips removed from solution for final observation.

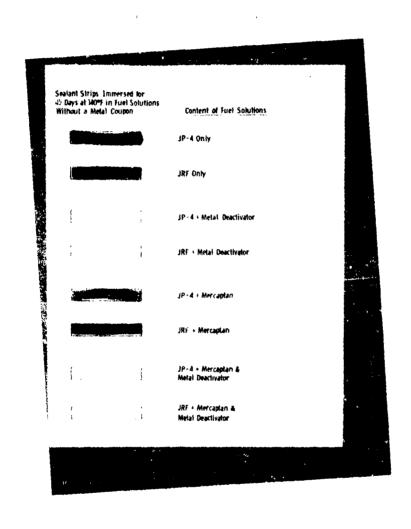
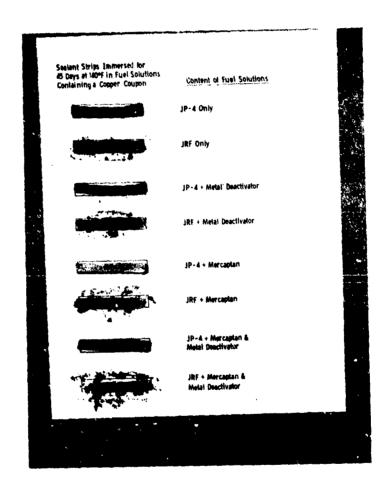


Figure 27. Control polysulfide sealant specimens for chalking tests.



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Figure 28. Polysulfide sealant chalking tests with metallic copper.

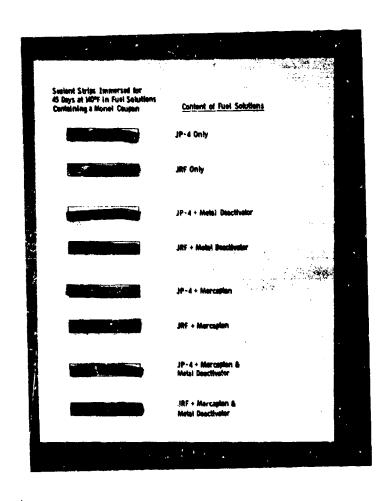
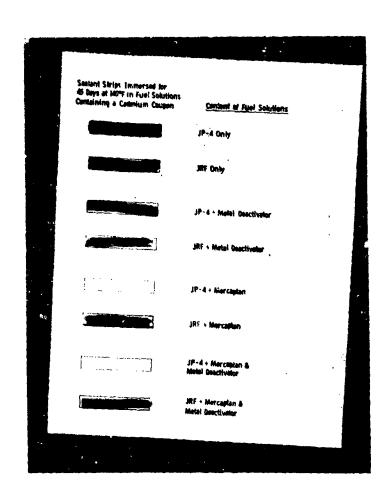
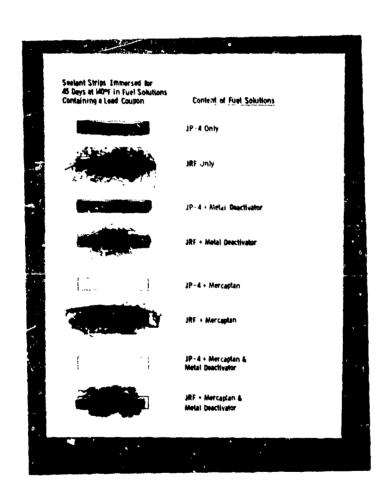


Figure 29 Polysulfide sealant chalking tests with Monel metal.



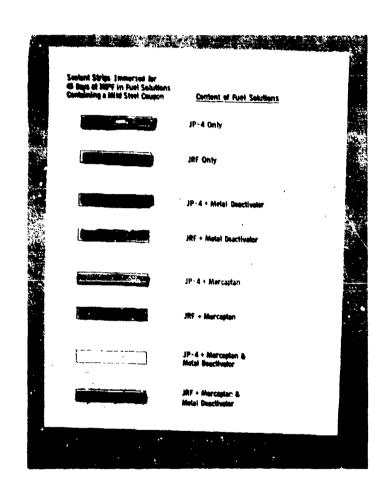
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Figure 30. Polysulfide sealant chalking tests with metallic cadmium.



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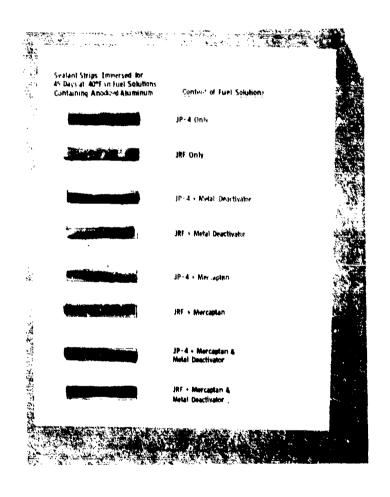
Figure 31. Polysulfide sealant chalking tests with metallic lead.



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Figure 32. Polysulfide sealant chalking tests with mild steel.



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Figure 33. Polysulfide sealant chalking tests with anodized aluminum.

mild steel and aluminum; but less severe with copper and lead. Chalking with cadmium was about the same in both fuels.

- (2) Sealant chalking in JP-4 occurred with all metals except lead, and it also occurred in JP-4 without metal. It is surprising that chalking occurred in the control JP-4 containing 400 ppb lead and not in JP-4 fuel with the lead coupon. The results make it appear that the presence of a lead coupon somehow acts as a chalking inhibitor. This phenomenon is probably effected by other unrecognized factors such as minor differences in sealant specimens.
- (3) Sealant chalking in JRF occurred with all tests except for those containing Monel, anodized aluminum and no metal (control fuel).

Summary

The fuel/metal equilibrium studies show that steady-state concentrations would be reached only after very long periods of time. However, the results of the sealant chalking studies indicate that only small quantities of some dissolved metals are enough to initiate severe cases of polysulfide sealant chalking. The presence of mercaptan accelerates the rate of chalking while the presence of the tested metal deactivator decreases chalking at a rate dependent upon the specific fuel/metal combination. The major factor determining sealant chalking appears to be the combination of a particular metal with a specific fuel.

4. POLYSULFIDE SEALANT CHALKING, FOLLOW-UP STUDY

The polysulfide sealant chalking results in the initial study were much more severe than had been expected. Particularly puzzling was the fact that sealant chalking occurred in baseline JP-4 but not in JRF fuel which contains mercaptan in its formulation. Additional studies were therefore conducted with the original and different batches of JP-4 and the original and another lot of sealant. No metal coupons or mercaptan were used in this study. Peroxide and metals analyses were conducted on all three JP-4 test fuels.

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Before the additional chalking studies with the baseline fuels were performed, additional studies with the metal deactivator N, N'-bis-salicylal-1,2-propane diamine were conducted. These tests were designed to show whether the deactivator was just as effective when applied to metal coupons before the chalking tests as when it was present in the jet fuel during the chalking tests.

Summary of Results

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- A. The original JP-4 used in the initial sealant chalking study was found to contain 54 ppm peroxides. Severe sealant chalking was again obtained with this fuel in the repeat studies with both lots of sealant, but no chalking was obtained with either sealant when two new batches of JP-4 containing little or no peroxides were used. The peroxide is strongly suspected to have caused the severe deterioration observed.
- B. The only metal found in any of the JP-4 test fuels was a small amount of lead, and this appears to some extent in all three fuels.
- C. The metal deactivator appears to lessen the severity of sealant chalking, as shown in the previous study. The deactivator

is not as effective when it is applied to metal coupons before the chalking tests as it is when it is present in jet fuel during the chalking tests.

Determination of Metal and Peroxide Content in Test Fuels

The original JP-4 used in the initial study, and two new batches of JP-4 labeled B-20 and F-2, were analyzed for metal content and peroxides. The metals were determined by emission spectrographic analysis and the peroxide analysis was conducted according to ASTM Method D 1563-60. Results are shown in Table 14 below.

TABLE 14. METAL AND PEROXIDE CONTENT OF FUELS

JP-4 sample	Peroxide content, ppm	Lead content, ppm	Other trace metals
Original	53.8	0.40	ND ^a
B-20	0.0	0.040	ND
F-2	0.5	0.125	ND

aND = None detected in low ppb range.

No zinc, aluminum, copper. tin, or iron was detected in the fuels at the low ppb levels.

Repeat Sealant Chalking Studies with Baseline Fuels

Sealant chalking studies were conducted for 45 days at 140°F with the three batches of JP-4 fuel and two batches of polysulfide sealant. The one lot of sealant had been used in the initial study and the second lot came from another Air Force contractor (University of Dayton Research Institute). No metal coupons, mercaptan, or metal deactivator were used in the studies. One 1/8" x 1/4" x 2" strip of sealant from each batch was vertically suspended in 500 milliliters of each batch of JP-4 for a period

of 45 days at 140°F. The strips were then removed from solution, dried with a paper towel, and evaluated for sealant chalking. A photograph of the tested strips is shown in Figure 34.

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Both batches of sealant showed severe chalking in the original JP-4 containing 54 ppm peroxide. Neither batch of sealant showed any chalking or deterioration in either the B-20 or the F-2 batch of JP-4. It is strongly suspected that the high peroxide level in the original test fuel is responsible for the severe sealant deterioration observed.

Other test data for the two additional batches of JP-4 were recorded within the Air Force Fuel Quality Laboratory and are presented in Table 15.

Alternate Method of Applying Metal Deactivator

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The initial sealant chalking study result showed that the metal deactivator, N,N'-bis-salicylal-1,2-propane diamine, lessened the severity of chalking in many cases regardless of whether caused by the presence of peroxide or mercaptan in the fuel. The metal deactivator was dissolved in the JP-4 at a concentration of 6 ppm for this study. It was then questioned whether the deactivator might be just as effective if it were applied to the metal surface prior to the sealant chalking study.

Four comparative sealant chalking tests were run with copper coupons, JP-4, 150 ppm butyl mercaptan (50 ppm mercaptan sulfur), and the metal deactivator. In two tests the butyl mercaptan was present and in the other two it was not. In one each of these two sets of tests, the metal deactivator was present in the JP-4 at a concentration of 6 ppm. In the other two tests, the copper coupon had been dipped into a 1% metal deactivator solution in cyclohexane prior to the chalking tests. The original JP-4 was used in the study since it was not known to contain peroxide at

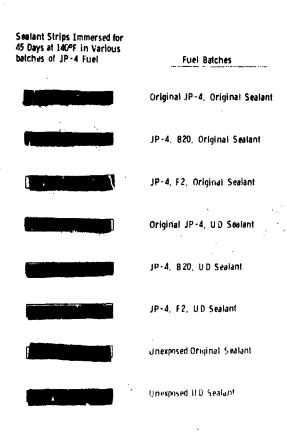


Figure 34. Polysulfide sealant chalking tests with various JP-4 specimens.

TABLE 15. ANALYTICAL TESTS ON NEW FUELS USED IN SEALANT TESTS a

	F-2	B-20
Total acidity, mg KOH/g	0.003	0.006
Aromatic content, vol %	1.0.7	9.3
Olefin content, vol %	1.6	1.9
Mercaptan sulfur, wt %	0.0000	0.0003
Total sulfur, wt %	0.04	0.02
API gravity, 60°F	54.4	55.6
Vapor pressure, psi	2.4	2.4
Freezing point, °F	-83	-82
Luminometer number	73	79
Smoke point	27.0	32.0
Copper strip, 2 hr at 212°F	la	1A
Existent gum, mg/100 mL	1.2	1.1
Anti-icing, vol %	0.13	0.00
Distillation		
IBP 10% recovered 20% " 50% " 90% " 95% " FBP	29 89 104 153 236 252 321	28 90 104 152 239 254 319

aMeasurements made by laboratory, Det 13SA-ALC/SFQLA, Aerospace Fuels Laboratory, WPAFB, OH 45433.

that time. The extent of sealant chalking in these tests at 140°F was monitored for a period of 18 days by observation through the walls of the glass test vessels, and after 100 days the sealant strips were removed from solution for a final evaluation. The results of these tests are shown in Table 16.

Applying the metal deactivator to the copper coupons prior to immersing in the JP-4 did not appear as effective in reducing sealant chalking as having the deactivator present in the JP-4.

SEALANT CHALKING VERSUS METHOD OF APPLYING METAL DEACTIVATOR® TABLE 16.

1% Deactivator pre-applied, 50 ppm sulfur in JP-4	No change	Yellowish grey	Yellow edges	Light grey, cracks	Chalked, the most deteri- orated strip, 1 crack almost thru entire thick- ness, many smaller cracks, very brittle
6 ppm Deactivator, 50 ppm sulfur ^b in JP-4	No change	Yellowish grey	Yellow edges	Yellow edges	Chalked, but no cracks
1% Deactivator pre-applied	No change	No change	No change	Yellow edges	Chalked, 2 cracks more than halfway thru sealant, few hairline cracks, noticeably brittle
6 ppm Deactivator in JP-4	No change	No change	No change	Yellow edges	Chalked, 3 cracks more than halfway thru sealant, 1 020 other surface cracks
Days	4	11	13	18	100°

This fuel was found to ^aSealant strips immersed in JP-4 solutions containing copper coupons at 140°F. contain 54 ppm peroxides and 0.40 ppm lead (only metal).

b As butylmercaptan.

^cStrips were removed from JP-4, wiped dry, and then observed.

5. INVESTIGATION OF PEROXIDE COMPOUNDS FORMED IN BASELINE JP-4 USED IN CHALKING STUDIES

Introduction

The baseline JP-4 used for sealant chalking studies in the previous test program (subsection 4) was found to cause severe sealant chalking on a polysulfide sealant. Subsequent fuel analyses showed that this JP-4 contained a high concentration of peroxides and acids. The purpose of this task was to isolate and identify the peroxides present in the JP-4, and determine whether there is a correlation between peroxide concentration and acid content (acid number).

Most of the JP-4 having a high peroxide content (176 ppm after 9 months storage) was consumed during the sealant chalking studies, and none was available for peroxide identification studies. This fuel had been drawn from a 35-gallon drum and then stored six months in a clear 5-gallon glass jug on a bench top prior to initial use. A new sample of fuel drawn from the 55-gallon drum was found to contain less than 2 ppm peroxides. Consequently, 1,500 milliliter quantities of the baseline JP-4 were exposed to a variety of environmental conditions in an attempt to generate enough peroxides for identification studies and acid number correlation.

Peroxide Formation and Correlation With Acid Number

A Navy report concerning the formation of organic peroxides in JP-5 (ref. 2) indicated that high temperature and presence of oxygen and water were the key factors leading to the rapid formation of peroxides in hydrocarbon fuel. Therefore, four 2-liter flasks containing about 1,500 milliliters of baseline JP-4 were connected to water-cooled reflux condensers and exposed to variations of these parameters. All flasks were injected with

free water and bubbled with air, but each flask was heated at a different temperature.

Flask No. 1 was heated to 190°C and after four hours the fuel had turned a brown color. Flask No. 2 was heated to 150°C and after five hours the fuel had turned an amber color. Both tests were discontinued when it was learned that fuel degradation was occurring without an increase in peroxide content. Flask No. 3 was heated to 65°C for a period of about two months and was periodically monitored for peroxide content and acid number. Flask No. 4 additionally contained a strip of polysulfide sealant and mild steel and was heated to 60°C, also for a period of about 2 months. The peroxide and acid numbers gradually increased in both flasks, but not at the rate expected and required. When it became evident that moisture, oxygen, and temperature were not the key factors for rapid peroxide formation in JP-4 fuel, a quart bottle of JP-4 was then placed on a bench top in front of an east window to investigate the possibility of peroxide formation by photolysis. The sealant/JP-4 was not exposed to direct sunlight, but it was now suspected that perhaps daylight may have been responsible for the high level of peroxide formation. This suspicion was confirmed when a significant quantity of peroxide began to form in a relatively short period of time.

All analysis results for peroxides and acid number are shown in Table 17. Peroxide concentration was determined by the iodide/sodium thiosulfate method described in ASTM D 1563-60. Total acidity was determined by ASTM Method D 3242.

The test results clearly indicated that visible light was the primary cause of peroxide formation in the JP-4 used for sealant chalking studies. The test results also showed that there is some correlation between peroxide content and acid number, even though the acid number data appeared to fluctuate.

TABLE 17. PEROXIDE CONTENT AND ACID NUMBER

	Exposure time	Peroxide content,	Acid number	Comments
Α.	JP-4 Stored in c	lear 5-gallon (Slass Jug and Used	for Chalking Studies
	0	1.1	0.003	
	6 months	53.8		
	9 months	176.0	0.101, 0.097	
в.	Clear Glass Bott	le of JP-4 in E	ront of East Wind	<u>on</u>
	0	2,2		
		10.9, 10.9	0.033	
	17 days	14.0	0.024	
_	_	•		
c.	Clear Glass Bott	le of JP-4 in a	Dark Cabinet	
	0	1.1	0.003	
	13 days	1.3	0.004	
	40 days	1.8	0.012	
	95 days	2.2	0.038	
	112 days	1.3, 1.0	0.010	
		_		
D.	Flask No. 1 - Ex	posed at 190°C	to Bubbled Air an	d Wree Water
	0	1.1	0.003	
	4 hours	∿1.1		Fuel turned brown
E.	Flask No. 2 - Ex	posed at 150°C	to Bubbled Air an	d Free Water
	Ū	1.1	0.003	
	5 hours	∿1.1		Fuel turned amber
F.	Flask No. 3 - Ex	posed at 65°C t	co Bubbled Air and	Free Water
	0	1.1	0.003	
	3 days	∿1.1		
	13 days	5.2		
	29 days	3.9	0.038	
	3.7 days	7.4	=++ ==	
	55 days	7.2	0.013	
G.	Flask No. 4 - Sea	alant and Mild	Steel Strips, Exp	osed at 60°C with Air & Water
	0	1.1	0.003	
	3 days	∿1.1		
	13 days	2.9		
	29 days	7.3	0.043	
	39 days	9.3		
	55 days	10.3	0.010	

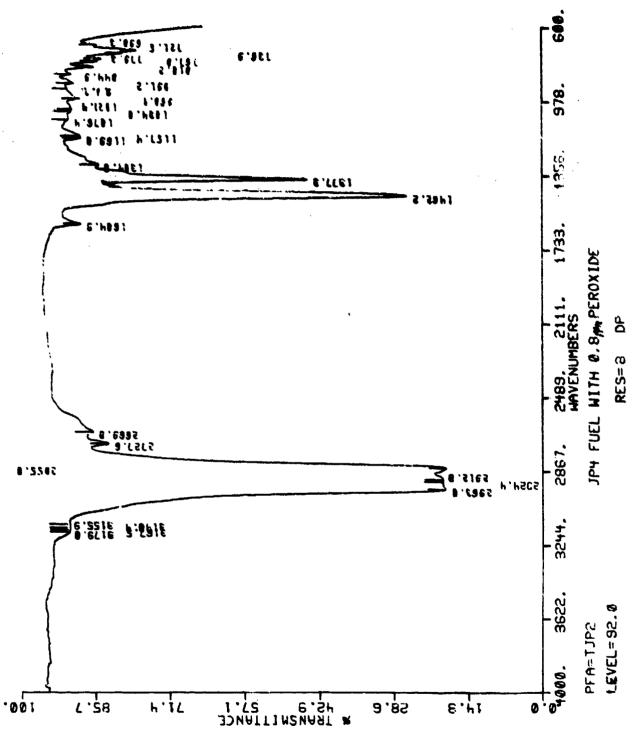
AND ENGLISHED STATES OF THE PROPERTY OF THE PR

Analytical Procedures

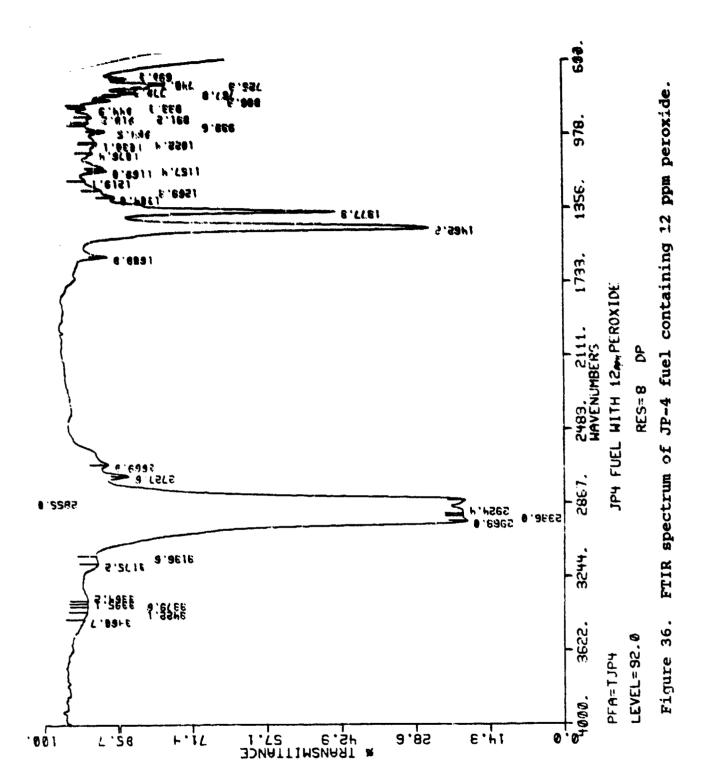
Because of the reactive nature of peroxides and hydroperoxides, care must be taken in the selection of an analytical method for their characterization. Mass spectrometry, for example, yields little direct information on these compounds even when low-voltage ionization techniques are employed. The Fourier-transform infrared technique provides a sensitive and flexible method of analysis, yet does nothing to promote further chemical reactions. A Digilab Model FTS-15 B/D system equipped with a General Data Nova 2/10 minicomputer was used for this work.

Fourier-transform infrared (FTIR) spectra were recorded for samples of baseline and peroxidized JP-4 fuels containing 0.8 ppm and 12 ppm of peroxide, respectively. The spectra shown in Figures 35 and 36 are the result of the co-addition of 100 scans of fuel in a 0.025 mm liquid cell, ratioed against a 50 scan reference. The spectra show that the peroxide-containing fuel had absorption bands at 3400, 1713, and 767 wave numbers (cm⁻¹), whereas the baseline fuel shows no absorption in these locations.

A computer subtraction of the spectrum of the baseline fuel from that of the peroxidized fuel is shown in Figures 37 and 38. In spectral subtraction, the larger the scaling factor (SCB), the greater the percent of the reference spectrum subtracted. The SCB is 0.8 for the spectrum in Figure 37. Figure 38, however, is a composite of 13 subtraction spectra with scaling factors ranging from 0.3 (top trace) to 1.5 (bottom trace). The persistence of certain bands even after obvious over-subtraction shows that the bands are, in fact, unique to the fuel having the high peroxide content. These bands are located at wave numbers 1713, 1126.5, 964.5, 875.8, 767.8, and 729.2. Their significance will be discussed under Results and Discussion.



FTIR spectrum of JP-4 fuel containing 0.8 ppm peroxide. Figure 35.





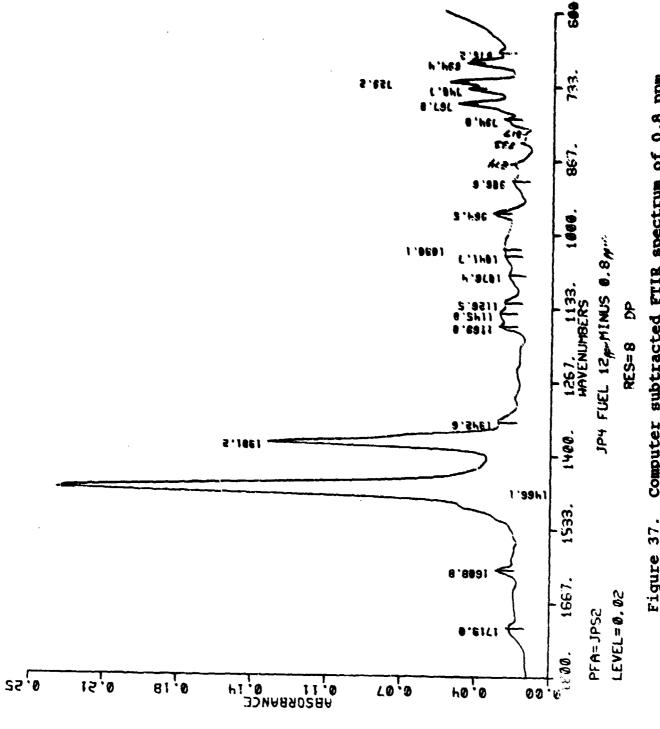
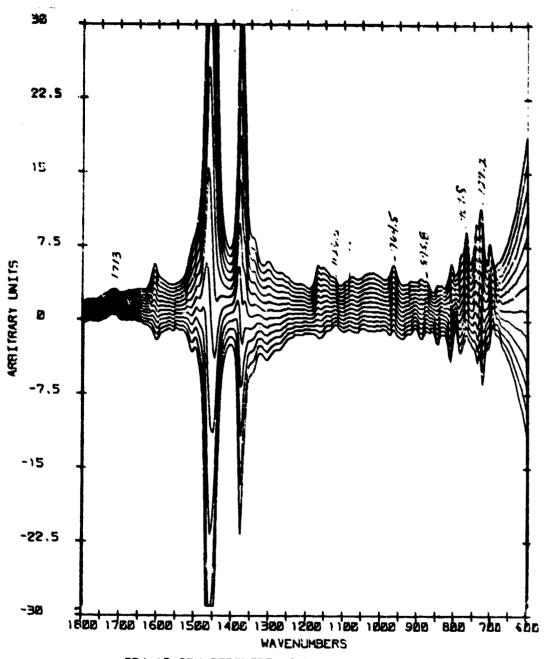


Figure 37. Computer subtracted FIIR spectrum of 0.8 ppm (peroxide) fuel from 12 ppm fuel.



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JP4 12 PPM PEROXIDE MINUS JP4 Ø.8 PPM PEROXIDE

 PFA=JP25
 PFB=JF24

 NSCANS=100
 RES=6
 DP
 NSCANS=100

 FCI=SB
 PFB=JF24
 NSCANS=100

Figure 38. Composite of 13 FTIR subtraction spectra with scale factors ranging from 0.3 (top trace) to 1.5 (bottom trace).

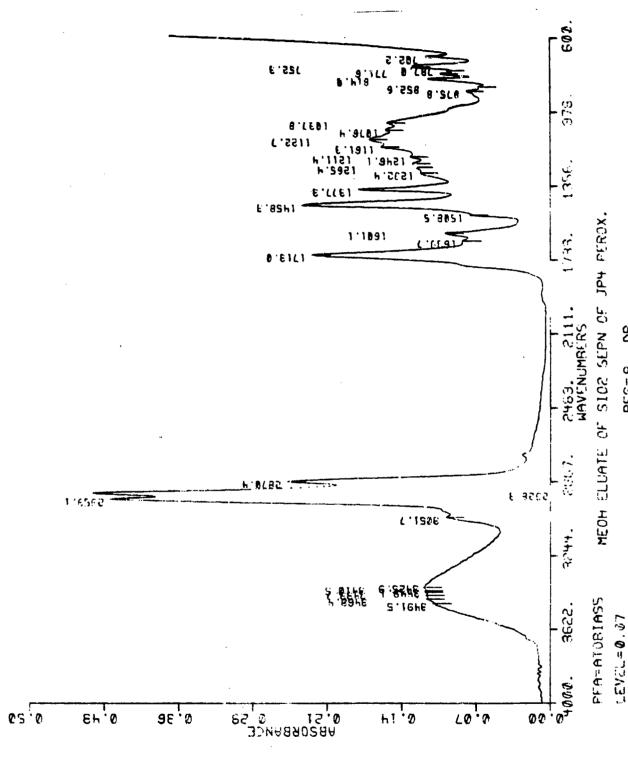
A 200 ml portion of JP-4, containing peroxides at the 32 ppm level, was passed through a silica gel column. The fuel was the same as that used for the earlier scalant chalking study. The column was eluted with methanol and a spectrum of the residue was recorded after evaporation of the methanol. To further separate the isolated materials, 1.08 g of this residue was applied to a fresh silica gel column and sequentially eluted with solvents as shown in Table 18.

TABLE 18. SEPARATION OF RESIDUE ISOLATED FROM TEST JP-4

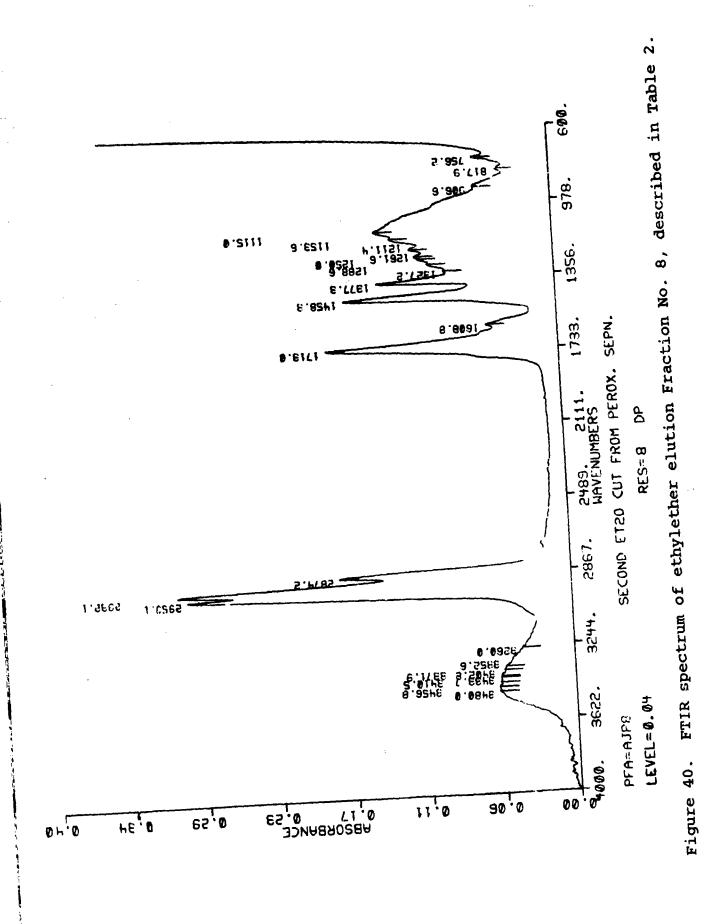
Fraction number	Elution solvent	Volume, ml	Weight residue, mg	Percent of total
1	Pentane - 1	50	0	C
2	Pentane - 2	50	0.3	0.03
3	Methylene chloride - 1	50	392.5	36.3
4	Methylene chloride - 2	50	51.4	4.8
5	Methylene chloride - 3	50	32.3	3.0
6	Methylene chloride - 4	20	9.3	0.9
7	Ethylether - 1	25	13.5	1.3
8	Ethylether - 2	2	146.2	13.5
9	Ethylether - 3	23	196.1	18.2
10	Acetone	50	~150	13.9
11	Methanol	50	~50	4.6
Total				~96. 8

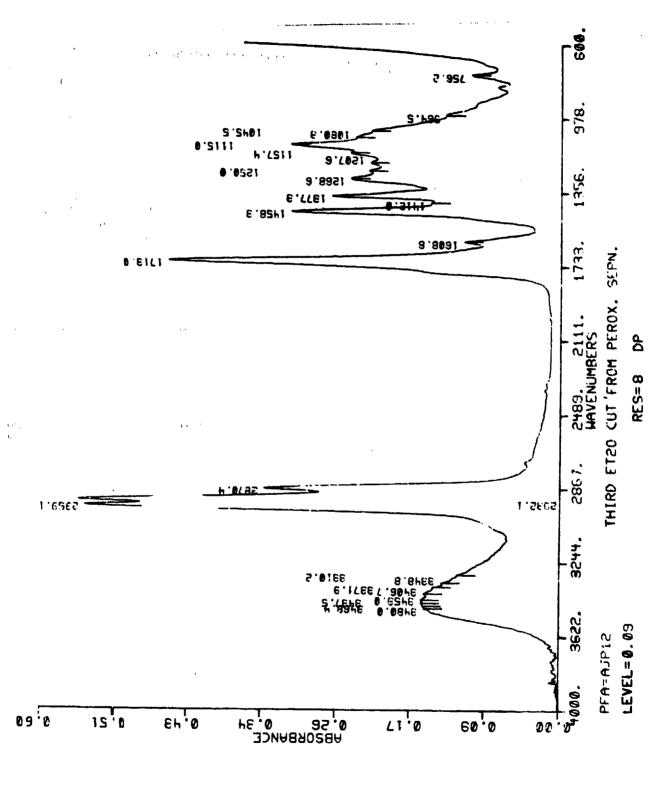
Infrared spectra were recorded for the initial residue (Figure 39) and each fraction after evaporation of solvent. The initial residue exhibited a strong carbonyl band at 1713 cm⁻¹ and other bands at 1126.5 and 875.8 cm⁻¹. These bands appeared for the eluted fractions 8, 9, and 10. Their spectra are shown in Figures 4C to 42. These three fractions showed a strong positive test (red color) for peroxides using the ferrothiocyanate test for peroxide.^a

This color reaction is the basis for ASTM Method D 1022, "Peroxide Content of Light Hydrocarbons." Though the reaction has a positive interference due to unsaturated acids, the formation of these compounds is unlikely.

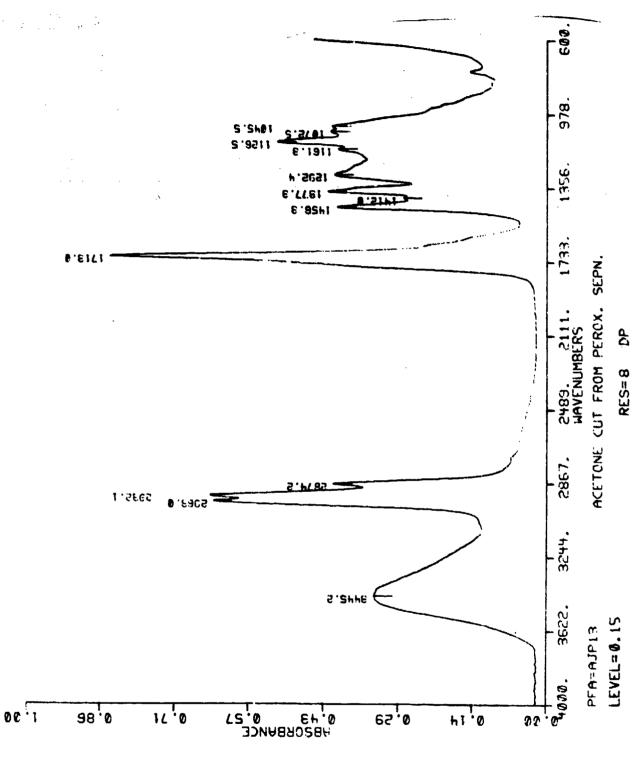


FTIR spectrum of residue from JP-4 containing 32 ppm peroxides, after passing through a silica gel column and being eluted with methanol. RES=8 Figure 39.





FIIR spectrum of ethylether elution Fraction No. 9, described in Table 2. Figure 41.



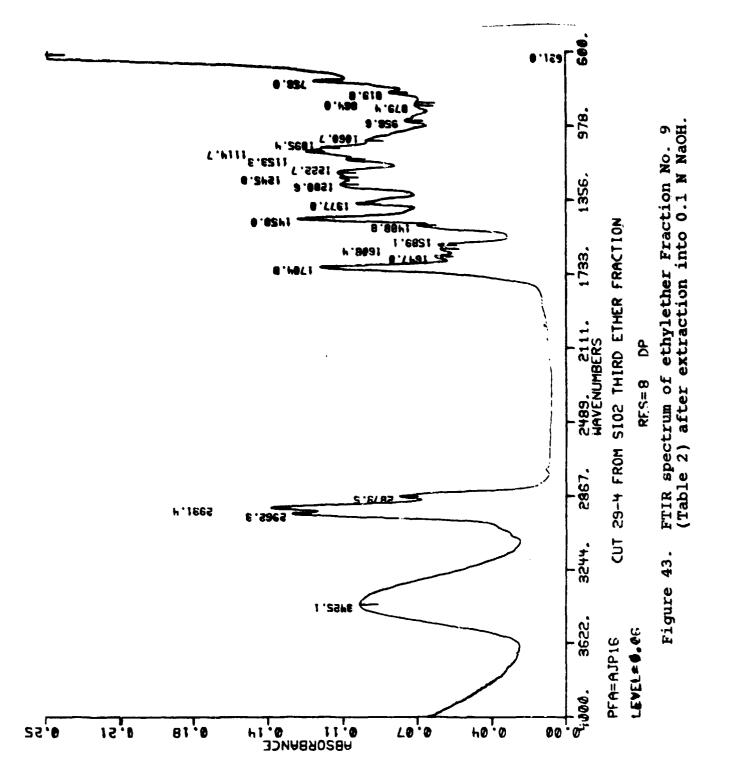
FIIR spectrum of acetone elution Fraction No. 10, described in Table 2. Figure 42.

The third ethylether fraction (No. 9) from the above separation was further separated by extraction with 3 ml of hexane, which essentially dissolved the residue. This hexane solution was extracted with 3 ml of 0.1 N NaOH solution at 0°C. The mixture was centrifuged to disperse the emulsion. The aqueous phase was separated from the hexane layer, neutralized with 0.1 N HCl, and re-extracted with 3 ml of hexane. The hexane was separated and evaporated in a flow of nitrogen to yield a 3.9 mg residue. The spectrum of the residue is shown in Figure 43.

Results and Discussion

Essentially the same spectral data were obtained for peroxide containing JP-4 samples whether the peroxides were formed accidentally, as in the previous sealant chalking study, or purposely formed by heating and bubbling with moist air. The high levels of peroxide obtained during the sealant study were never realized when deliberate peroxide generation was attempted. The techniques suggested in the Naval Air Propulsion Center report of reference 2 were employed as well as simulation of chalking test conditions, even to the point of adding a metal coupon and sealant to the fuel. Despite the Naval report suggestion that brown bottles be used for the peroxidation, it was felt that the peroxide levels achieved previously were possibly related to light intensity. Test confirmed this to be true. In a short time, fuel exposed to light from a window developed a peroxide level higher than that produced by any other technique.

Differential IR absorption bands, obtained through spectral subtraction, are localized in three of the eleven fractions from a liquid chromatograph separation of polar fuel constituents. These bands represent the compositional differences between the baseline fuel and that same fuel after being subjected to oxidizing conditions. Important features of the spectra for these three fractions are a carbonyl absorption at 1713 cm⁻¹, lack of aromatic



character as indicated by the absence of a band at approximately 3000 cm $^{-1}$ and the appearance of a hydroxyl (OH) band at ~ 3400 cm $^{-1}$. Other bands indicate aliphatic character. These data suggest the presence of aliphatic ketones and alcohols. The three fractions gave a strong positive color reaction for the peroxide test using the ferrothiocyanate reagent. Absorption bands for organohydroperoxides were not prominent in the spectra, suggesting that the loss of some hydroperoxide through a reduction reaction had occurred. Spectra show essentially a mixture of aliphatic alcohols These ketones and alcohols are common products from the air oxidation of aliphatic hydrocarbons being formed both directly during the oxidation process and from the decomposition of hydroperoxides (ref. 2). The presence of a small amount of an organic acid was shown by the shift in the carbonyl band (ionized carboxyl) of the material extracted into 0.1 N NaOH. extraction effectively separated the acid materials from the ketones.

Somewhat surprisingly, there was no evidence that peroxides or hydroperoxides were formed from aromatic fuel constituents, even though these constituents are generally more subject to peroxidation. The fact that a variety of materials can serve as oxidation catalysts or initiators, even though present only at trace levels, contributes to the unpredictability of the peroxidation reaction.

The significance of acid number in the peroxide forming studies was not clear. A correlation exists between acid number and peroxide content but the values were not directly proportional.

Conclusions

Peroxides and hydroperoxides were formed in the subject JP-4 fuel by a photolysis reaction. These compounds were of an aliphatic nature, consisting largely of ketone and alcohol types. A small amount of organic acid was also found to be present. No evidence of aromatic peroxide formation was found.

6. EVALUATION OF THE MICROSEP-II WATER SEFAROMETER

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All aviation turbine fuel specifications include a requirement concerning the ease with which the fuel will release entrained or emulsified water. The water separation index, modified (WSIM) test, which is described in ASTM procedure D 2556, provides a numerical rating for the fuel's water separation characteristics.

The WSIM value of a fuel is measured with a device in which a water-fuel emulsion is prepared and metered through a cell containing a standardized glass-fiber coalescer. The cell effluent turbidity, caused by entrained water, is determined by light transmission through the fuel to a photocell. The output of the photocell is measured by a meter having a 0-100 scale, thus providing a numerical rating for the fuel. The original apparatus for the test, known as ASTM-CRC Water Separometer (Emcee Electronics, Inc.), has three functions: emulsion preparation, coalescence, and analysis.

The WSIM of a fuel can also be measured by other instruments, such as the Minisonic Separometer and the Microsep Separometer. Both units are manufactured by Emcee Electronics, Inc. The ASTM-approved Minisonic Separometer is a small-scale device using the same principles as the ASTM-CRC Water Separometer. The Microsep Separometer incorporates solid-state design and a self-contained power source, but it is not yet approved by the ASTM.

The purpose of this study, which was part of a program sponsored by ASTM Committee D-2, was to evaluate the Microsep unit by comparing its results with those of the Minisonic system. Table 19 lists some differences between the two instruments.

COMPARISON OF MINISONIC AND MICROSEP SEPAROMETERS TABLE 19.

Feature	,	
44	FILIT BUILD	Alcrosep
Portability	Requires a.c. electric power; can be hand carried	Internal rechargeable battery; can be hand carried (lighter)
Temperature range	65°F to 85°F	65°F to 85°F
Operation	Requires external timer; about 5-10 min per test; can be used multibatch	Programmed time (internal); 3.5 min per test (after preparation)
Emulsifier	Ultrasonic adjustable (timer and intensity) prescribed by ASTM, 25 sec ±1	Stirring (high speed) motor, programmed time (set)
Syringe drive	Time adjustable (prescribed by ASTM, 45 sec ±2)	Time (programmed-set)
Settling period	<pre>l min (prescribed by ASTM, operator timed)</pre>	l min (programmed-set)
Turbidity	Compares (split beam) original sample to filtered sample	Compares original sample (100% transmission in memory) to filtered sample (0-100 scale reading) transmission
Calibration	Field	Factory
Small parts	Kit supplied by EMCEE	Essentially same for each

Procedure

The preparation and procedure for the Minisonic Separometer as described in ASTM D 2550-74, Section A6 was followed in detail. For the Microsep system, a procedure contained in the operation manual furnished with the apparatus by Emcee Electronics, Inc. was followed. Tests were conducted as specified in the instructions (see Table 20) provided by the ASTM program coordinator. Data for a JP-4 test fuel are presented in Tables 21 and 22.

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Discussion

It was observed that if the sample vial is taken from the Microsep turbidimeter directly after reading and is placed in the Minisonic unit with the meter nulled using the appropriate blank, one gets consistently higher readings at the low end of the scale than with the Minisonic unit. This is shown in Table 23.

Several runs on each instrument were timed, and the timed operations were within the specified limits, as shown in Table 24.

Precision criteria for judging the acceptability of results are defined by a graph contained in ASTM D 2550 and shown in Figure 44. Repeatability is a measure of the variance in duplicate determinations by the same operator. For this evaluation, reproducibility is considered to be the difference in results from each of the two instruments using the same type of coalescence cell.

Reproducibility and repeatability of the data by these definitions are presented in Table 25.

Only 4 of 28 samplings failed to meet the repeatability test and 3 of these occurred with the Microsep Separometer. Three of the fourteen samplings failed the reproducibility test.

TABLE 20. TEST PROGRAM FOR MINISONIC AND NICROSEP-II SEPAROMETERS

I. TEST APPARATUS

- A. D 3602-77 Mini-Sonic Separameter B. MICHOSEP-11 (Modified by Rance Electronics)

II. TEST FLUIDS

	Jet A Blo	mda		Jet 3	?lends
Puel No.	Grac. of Additive	Amt. of Concentrate Par L of Fuel	Nuel,	Conc. of Additive	Amt. of Concentrate Per L of Fuel
JA.	**	***	77	***	
2 A	0.2 mg/L AOT	0.2 mL	28	0.6 mg/L AOY	0.6 mL
34	0.6 mg/L AOT	0.6 mL	38	1.2 mg/L MOT	1.2 mL
44	4 PTB DC1-4A	11 mL	43	4 PTB DCI-4A	11 mL
5A	6 PTB E-515	17 mL	5B	8.5 PTB B-515	24 mL
64	6 PTB E-515 0.5 ppm ASA-3	17 mL	63	8.5 PTB E-515 0.7 ppm ASA-3	24 mL
78	0.5 ppm ASA-3	0.5 mL	78	0.7 ppm ASA-3	0.7 ml.

111. PROGRAM

- A. Prepare reference fuels in accordance with D 3602, Appendix A-2. If tests of Blends 2 and 3 fail to meet the criteria of D 3602, Tables 1 or 2, use a fresh reference fuel.
- B. Prepare Fuel Blends in accordance with II above by adding concentrate (1000 mg/L of each additive) to reference fuel.
- C. Run tests using Plasticels by both MSS and Microscp, and enter results on attached Data Sheet.
- D. Repeat each Fuel Blend with the same operator on the same day in random order.
- E. Bun all blends using the Metal Cell to compare with plasticels. (Sufficient additive concentrate has been furnished for this purpose. Many laboratories prefer to use Netal Cells for economy runsons.)

TABLE 21. DATA SHEET FOR MICROSEP-II - MSS PROGRAM

DATA SHEET (Complete RF lype and Blends For Attachment I Test Fluid Table)

TESTING LABORATORY _____ AF APL NET FUEL MASK JET JP.4 COALESCER CELL TYPE METAL CELL WITH EMCEE PART 1104-19 COALESCER DISCS Mand

			A SSK	uting	Microser	Rat Inc
No.	则2 4.	Addition	Test 1	Test 2	Tri.1	Test 2
1		Hone	100	99	100	100
2	2.6	TOA	75	72	62	65
3	1.2	AOT	43.	67	_5.8	<u>55</u>
4	16	BC3-4A	95	9_8	15	90
5	24	HITEC E-515	97_	18	96	95
6	**	HITEC E-515 ASA-3	93	9/	85	89
7	e. 7	C-AZA	97	97	9.3	9/

CONTENTS: # ADDITIVE 6 PTB -E-515 }

O.5 PPM ASA-3 }

FURNISHED

24 ML/LITER FUEL WAS USED

TABLE 22. DATA SHEET FOR MICROSEP-II - MSS PROGRAM

DATA SHEET (Complete RF Type and Blends Per Attachment I Test Fluid Table)

		COALESCER CRUI	Tyre Plas	TICEL	EMCEE PART	59/0A
			MSS R	ating_	Merosep	Kating
<u> No.</u>	mg/1.	Additive	Test 1	Test 2	Test 1	Test 2
1		None	100	79	100	99
2	<u>6.6</u>	TOA	92	_77_	64	66
3	1.2	AOT	76	67	49	50
4	11	DC1-4A	14	83	95	85
5	24	HITEC E-515	98	98	87	90
6	*	HITEC E-515 ASA-3	95	7/	89	82
7	<u>0.7</u>	C-AZA	47	97	88	88
C0%	ents :	* 6 PTB 0.5 PPM	E -515	3	FURNISHED	

BARGer Lles Signature

TABLE 23. TURBIDITY MEASUREMENTS ON THE SAME SAMPLE FOR FACH TEST INSTRUMENT

Sample	Microsep reading	Minisonic reading
1B,	300	100
1B ₂	99	99
2B,	66	79
2B ₂	62	72
3B ₁	88	92
3B ₂	50	74
4B ₁	85	90
4B2	95	96
5B ₁	90	96
5B.	96	98
6B ₁	82	91
6B ₂	85	91
7B ₁	88	93
7B2	93	97

TABLE 24. TIMING OF SEQUENTIAL EVENTS DURING SEPAROMETER OPERATION

		T	ime,	secon	ds		_
	Micro	sap r	unsa	Mini	sonic	runs	_
Event	#1	#2	#3	#1	#2	#3	(ASTM D 2550)
Emulsification (duration)	28	29	31	26	25	25	25 ± 1
Extraction (duration)	44	45	44	44	47	45	45 ± 2
Elapsed time at reading	188	189	189	180	183	190	-
Settling (duration)	60	60	60	61	60	60	60

Time cycle automatically controlled.

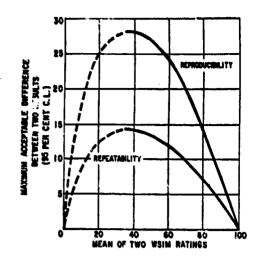


Figure 44. Variation of repeatability and reproducibility with rating level (taken from ASTM D 2550).

TABLE 25. REPRODUCIBILITY AND REPEATABILITY OF PERCENT TRANSMISSION MEASUREMENTS FOR THE TWO SEPAROMETERS

		Mini	sonic				Micro	gsap	
Sample	High	Low	Avg	Repeat- ability Difference	High	Low	Avg	Repeat- ability Difference	Reproduci- bility Difference
				Plast	ticels				
1 2 3 4 5 6 7	100 82 76 84 98 95	99 77 67 83 98 91 97	99.5 79.5 71.5 83.5 98 93	1 5 9 1 0 4 ປ	100 66 50 95 90 89 88	99 64 49 85 87 82 88	99.5 65 49.5 90 88.5 85.5	1 2 1 10 3 7	1 18 27 12 11 13
•	31	31	31		l Cells		80	Ū	9
1 2 3 4 5 6 7	100 75 67 96 98 93 97	99 72 63 95 97 91	99.5 73.5 65 95.5 97.5 92	1 3 4 1 1 2 0	100 65 58 95 96 89	100 62 55 90 95 85 91	100 63.5 56.5 92.5 95.5 87 92	0 3 3 5 1 4 2	1 13 12 6 3 8 6

One of the major differences in the operation of the Minisonic and Microsep Separometers is in the emulsification cycle. In operation it appears the mixing is much more vigorous and the mixing time is slightly longer in the Microsep unit. Data show the ratings by the Microsep unit to be lower than those by the Minisonic. Table 23 indicates that the difference is in the turbidity measuring portion of the operation rather than in the emulsification step. The turbidity readings obtained from the Microsep unit are lower than those from the Microsep unit are lower than those from the Microsep unit are lower than those from the Minisonic.

At present there is no field method for calibrating or checking calibration of the Microsep Separometer. Section A2.2.7.2 of the Minisonic Separometer Operation Manual describes a method of calibration. A method similar to this for the Microsep unit would be useful at least to check the calibration even if there is no means of field calibration.

7. ANALYSIS OF RAMAN SPECTROSCOPY DATA FOR GOODNESS-OF-FIT TO FOUR STATISTICAL DISTRIBUTIONS

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As a part of the error propagation analysis task, several sets of Coherent Antistokes Raman Spectroscopy (CARS) data have been analyzed for goodness-of-fit to four statistical distributions. This was accomplished by utilizing a goodness-of-fit program on the MRC computer. The program attempts to fit each data set to each of four theoretical distributions, namely, Weibull, normal, gamma and log-normal distributions.

Output of the computer program provides parameters that sufficiently describe the distribution, such as the mean and standard deviation for the normal distribution. Other parameters are given that describe the goodness-of-fit for a particular distribution. Some of these parameters are skewness, kurtosis, and a chi-square value. Additionally, the theoretical and actual frequencies of data groups in each class interval are provided.

Skewness is a measure of symmetry. A value of zero indicates left-to-right symmetry. A positive value indicates skewness to the right; that is, the tail of the distribution extends further to the right as the positive number becomes greater in magnitude. A negative number indicates skewness to the left in a similar fashion.

The quantity kurtosis is a measure of peakedness or thinness of a distribution. A value of three is the perfect shape of a normal distribution. Values greater than 3 indicate a tall peak and narrow width of the distribution, smaller values indicate a shorter, broader peak.

To determine how well our data fit a particular statistical distribution, a chi-square goodness-of-fit test can be used. The value of chi-square is calculated as follows:

Chi squared,
$$\chi^2 = \sum_{i=1}^{k} \frac{\left[O(n_i) - E(n_i)\right]^2}{E(n_i)}$$

where $E(n_i)$ is the expected frequency, $O(n_i)$ is the observed frequency, and n_i is the number of observations in the ith cell or class interval. A "perfect fit" yields a χ^2 value of zero. As the magnitude of χ^2 increases, the likelihood that the theoretical distribution fits the data diminishes.

The procedure for using the chi-square test involves hypothesis testing. The null hypothesis H_0 and its alternative H_1 are stated:

 ${\rm H}_{\rm O}$ - These data fit the theoretical distribution

 H_1 - The data do not fit the distribution

A good fit leads to the acceptance of H_0 , whereas a poor fit leads to its rejection. For this test, we must also define a level of significance, α , and a critical value, χ_{α}^2 , from the chi-square table. The α and χ_{α}^2 determine the critical region $\chi^2 > \chi_{\alpha}^2$, i.e., our calculated χ^2 exceeds the table χ_{α}^2 . If, indeed, our chi-square value exceeds the table values, we reject the null hypothesis and conclude that our data do not fit the theoretical distribution we are considering.

An example is taken from a set of data for a still air sample at 24.5 inches. A chi-square value of 15.5 with 4 degrees of freedom is given for the normal fit. Using the chi-square distribution shown in Table 26 (ref. 3), the 0.05 column with 4 degrees of freedom gives a critical value of 9.49. We must reject the null hypothesis and repeat this test for other distributions. Now, if one traverses the table to the right, under A = 0.01, χ^2 critical = 13.28. From the example cited above of where the chi-square test value = 15.48 with 4 degrees of freedom, we can say that we reject the null hypothesis (at 5% level) but cannot reject it for the Weibull Maximum Likelihood, the gamma, or the log-normal distributions.

However, if we relax the requirement to 0.1 of making a Type II error, we see that all distributions must be rejected except the log-normal. One can then conclude that the data may be roughly log-normal. Table 27 is a summary of the results.

8. ANALYSES TO DETERMINE CAUSE OF PLUGGING IN MAIN ENGINE FILTERS

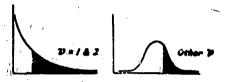
The main engine filter, designed to remove solid contaminants from jet fuel before it reaches the engine, was found to be plugging in certain aircraft. When the plugged filters were removed, it was observed that the contamination was not uniformly distributed. A small but thick mat of fiberous material was deposited near the

TABLE 26. CHI-SQUARE DISTRIBUTION

Chi-Square Distribution

A denotes the right tail area for the values of χ^2 given below. ν denotes the number of degrees of freedom (df).

関連が対象の関連を対象を対象した。



r or df	A = 0.99	A = 0.98	A = 0.95	A = 0.90	A = 0.80	A = 0.70	A = 0.50
<u> </u>	.00014	.00043	.0039	.016	.064	.15	.46
2	.02	.04	.10	.21	.45	.71	1.39
3	.12	.18	.35	.58	1.00	1.42	2.37
4	.30	.43	<i>.</i> 71	1.06	1.65	2.20	3.36
5	.55	.75	1.14	1.61	2.34	3.00	4.35
3	.87	1.13	1.64	2.30	3.07	3.83	5.35
7	1.24	1.56	2.17	2.83	3.82	4.67	6.35
	1.65	2.03	2.73	3.49	4.59	5.53	7.34
9	2.09	2.53	3.32	4.17	5.38	6.39	8,34
10	2.56	3.06	3.94	4.86	6.18	7.27	9.34
11	3.05	3.61	4.58	5,58	6.99	8.15	10.34
12	3.57	4.18	5.23	6.30	7.81	9.03	11.34
13	4.11	4.76	5.89	7.04	8.63	9.93	12.34
14	4.66	5.37	6.57	7.79	9.47	10.82	13.34
15	5.23	5.94	7.26	8.55	10.31	11.72	14.34
16	5.81	6.61	7.96	9.31	11.15	12.62	15.34
17	6.41	7.26	8.67	10.08	12.00	13.53	16.34
18	7.02	7.91	9.39	10.86	12.86	14.44	17.34
19	7,63	8.57	10.12	11.65	13.72	15.35	18.34
20	8.26	9.24	10.85	12.44	14.58	16.27	19.34
21	8.90	9.92	11.59	13.24	15.44	17.18	20,34
22	9.54	10.60	12.34	14.04	16.31	18.10	21.34
23	10.20	11.29	13.09	14.85	17.19	19.02	22.34
24	10.86	11.99	13.85	15.66	18.06	19.94	23,34
25	11.52	12.70	14.61	14.47	18.94	20.87	24,34
26	12.20	13.41	15.38	17.29	19.82	21.79	25.34
27	12.88	14.12	16.15	18.11	20.70	22.72	26.34
26	13.56	14.85	16.93	18,94	21.59	23.65	27.34
29	14.26	15.57	17.71	19.77	22.48	24.58	28.34
30	14.95	16.31	18.49	20.60	23.36	25.51	29.34

(continued)

TABLE 26 (continued)

Chi-Square Distribution

r or df	A = 0.30	A = 0.20	A = 0.10	A = 0.05	A = 0.02	A = 0.01	A = 0.001
1	1.07	1.64	271	3,84	5.41	6.64	10.83
2	2.41	3.22	4.60	5.79	7.82	9.21	13.82
3	. 3.66	4.64	4.25	7.82	9.84	11.34	16.27
4	4.88	5.99	7.78	9,47	11.67	13.26	18.46
5	6.04	7.29	9.24	11.07	13.39	15.09	20.52
6	7.23	8,54	10.64	12,59	15.03	16.81	22.46
7	8.38	9.80	12.02	14,07	16.62	18.48	24.32
8	9.52	11.03	13.34	15.51	18.17	20.09	26.12
•	10.66	12.24	14.68	16.92	19.68	21.67	27.88
10	11.78	13.44	15,97	18.31	21.16	23.21	29.59
11	12.90	14.63	17.28	19.68	22.62	24.72	31,26
12	14.01	15.81	18.55	21.03	24.05	26.22	32.91
13	15.12	14.98	19.81	22.36	25.47	27.69	34.53
14	16.22	18.15	21.04	23.68	26.87	29.14	36.12
15	17.32	19.31	22.31	25.00	28.26	30,58	37.70
16	18.42	20.46	23.54	26,30	29.63	32.00	39.25
17	19.51	21.62	24.77	27.59	31.00	33.41	40.79
18	20.60	22.74	25.99	28.87	32.35	34.80	42.31
19	21.69	23.90	27.20	30.14	33.69	36.19	43.82
20	22.78	25.04	28.41	31.41	35.02	37.57	45.32
21	23.86	26.17	29.62	32.67	36.34	38.93	46.80
22	24.94	27.30	30.81	33.92	37.66	30.29	48.27
23	26.02	28.43	32.01	35.17	38.97	41.64	49.73
24	27.10	29.55	33.20	36.42	40.27	42.98	51.18
25	28.17	30.48	34.38	37.6.	41.57	44.31	52.62
26	29.25	31.80	35.56	38.88	42.86	45.64	54.05
27	30.32	32.91	34.74	40.11	44.14	46.96	55.48
28	31.39	34.03	37.92	41.34	45.42	48.28	56.89
29	32.46	35.14	39.09	42.56	46.69	49.59	58.30
30	33.53	36.25	40.26	43.77	47.96	50.89	59.70

	TABLE 27.	CALCULATED C	CHI-SQUARE VAI	VALUES	
	Weibull Maximum	Weibull			, <u>-</u>
Data Set	Likelihood	Squares, Y	Normal, Y	Gamma, Y	Normal, Y
Still Air Sample 24.5	7.94, 4	11.68, 4	15.48, 4	7.66, 4	3.9, 3
Air Ref.		4.94, 4	5.29, 4	4.75, 4	5.92, 3
	5.41, 3	5.6, 3	9.0, 2	3.94, 3	2.67, 3
	4.51, 1	_	5.91, 2		2.4, 2
Flame Ref. 24.5	3.89, 1		4.1, 1	1.34, 0	0.91, 1
Flame Ratio 24.5	1.4, 1	0.35, 0		•	0.43, 1
Samp	5.5, 3	5.4, 3		4.36, 4	3.6, 4
Vacuum Ref. 24.5	4.2, 3	4.4, 3		7.6, 3	7.9, 4
24.	6.7, 4	7.2, 3	13.0, 3	4,	2.4, 4
at	3.2, 1	2.5, 1		2.3, 0	3.8, 0
at 2.	0.9, 1	0.8, 1	0.9, 1	0.2, 1	0.6, 1
Air Flow Ratio at 2.75	5.6, 1	6.5, 1	5.5, 1	4.4, 1	2.9, 1
at 2.75	1.5, 1	2.0, 1	1.2, 1	2.6, 1	2.5, 1
Ref. at 2	0.12, 1	0.13, 0	1,	0.1,0	0.6, 0
		2.2, 1	.3,		
Flame at 8.4		1.73, 0	.5,	1.4, 0	1.4, 0
Flame Ref. 8.4			.5,	•	2.6, 1
Flame Ratio 8.4		5.1, 0	4.1, 0	4.7, 0	1.9, 0
Air at 14.5		2.5, 2		0.85, 1	
Air Ref. 14.5		1.0, 1	1.0, 2	0.46, 1	1.87, 2
Air Ratio 14.5		7.1, 0	6.3, 0		6.1, 0
Flame at 14.5	0.1, 0	0.02, 0	0.96,0	0,0	0.57, 1
Flame Ref. 14.5	1.85, 1	1.2, 0	1.7, 1	0.76, 1	1.2, 1
Flame Ratio 14.5	•	ı	1	ı	1.4, 1
Vacuum at 19.5	4.5, 3	4.6, 3	5.5, 3	4.3, 3	4.2, 4
Vacuum Ref. 19.5	4.1, 4			7.7, 4	9.0, 4
Vacuum Ratio 19.5	1.9, 3			1.5, 4	4.5, 4
Still Air 19.5	9.2, 3	8.2, 3	6.2, 2	6.1, 3	2.7, 4
Still Air Ref. 19.5	2.7, 4	3.6, 4	3.4, 4	2.5, 4	2.8, 4
					- 1

inner corners of the filter pleats and a fine particulate layer was spread over much of the paper surface. The large debris appeared to be a mixture of cellulose and synthetic fibers plus pieces of fuel tank foam. These materials, however, were not specifically characterized. The composition of the fine particulate residue was unknown, but it appeared to be responsible for the filter plugging that resulted in a high pressure drop. The main purpose of this work was to identify the fine particulate contaminate on these submitted filters labeled 77-0262, 77-0248, and 77-0270.

Analysis of Fine Particulate Residue

The fine particulate residue was scraped from the surface of the three filters and analyzed by emission and infrared absorption spectral techniques. The results for the semiquantitative emission spectrographic analyses are shown in Table 28, while a typical infrared absorption spectrum for the sample is shown in Figure 45.

The emission spectrographic analyses suggest that much of the particulate contamination may be characterized as terrestrial dust or dirt. This is indicated by the presence of significant quantities of aluminum, silicon, calcium, magnesium, and iron. The significance of the presence of cadmium and zinc is not known.

The IR absorption spectra indicate the presence of oxygencontaining compounds and low levels of hydrocarbons. The hydrocarbon bands are probably due to residual fuel, while the main
oxygen species are probably metal oxides. The presence of
inorganic carbonate is also suggested by a weak band at approximately 7 microns. The presence of some cellulose from scraping
the filter paper is indicated at a low concentration. These
spectral features are consistent with the characterization of the
contaminants as entrapped ambient dust.

TABLE 28. EMISSION SPECTROGRAPHIC ANALYSIS RESULTS FOR FILTER PAPER RESIDUES

	Percent	Concentrations for Followin	ng Samples
Element	77-0262	77-0248	77-0270
5.2	1.0	••	
Aluminum	>10	>10	>10
Silica	1-10	1-10	110
Calcium	1-10	1-10	1-10
Magnesium	0.5	0.3	1-10
Iron	0.5	1-10	1-10
Cadmium	ND*	0.4	1-10
Zinc	Ü.5	0.5	1-10
Chromium	0.1	0.2	0.5
Sodium	0.03	0.04	0.4
Manganese	0.05	0.05	0.2
Copper	0.3	0.1	0.2
Lead	G.Q5	0.05	0.2
Nickel	0.005	0.2	0.03
Titanium	0.05	0.4	0.1
Molybdenum	0.05	0.05	0.04

ND* - not detected

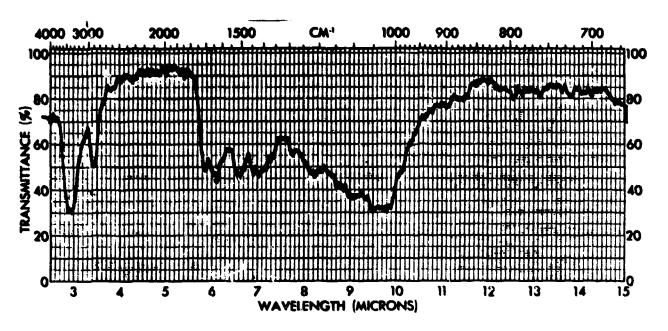


Figure 45. Infrared absorption spectrum of fine particulate residue.

Analysis of Filters for Impregnated Organic Material

It was suggested that organic material impregnated in the filter, rather than matter coated on its surface, might be responsible for the increased resistance to flow. To test this theory, the filters were extracted with a chlorinated solvent which was then analyzed for organic content.

The filter paper segments used for the previous analyses were extracted with methylene chloride at room temperature. The paper was cut into irregular shaped pieces for this purpose, with dimensions being roughly 1 x 1/2 inches. The methylene chloride extract was allowed to evaporate to near dryness in an evaporating dish and then the last several milliliters were transferred dropwise to a rock-salt plate where the remaining solvent was removed by further evaporation. An infrared (IR) adsorption spectrum of the residue was recorded.

Methylene chloride extractions were conducted on two different filters with approximately twice as much filter paper being used for the second extraction. No residue was visually observed on the rock-salt plate from either of the extractions, and no organic compounds were detected by the IR analyses. The IR spectrum for the extracts showed only the presence of water as indicated by bands at approximately 3 and 6 microns. This doubtlessly resulted from condensation during the cooling that was associated with the evaporation of solvent. Considering the high sensitivity of the infrared spectrophotometric technique for organic analysis and the excellent solvent properties of methylene chloride for most organic compounds, it can be concluded that no significant amount of organic material was contained on the filter as received.

9. TRACE METAL ANALYSES AND IDENTIFICATION OF PARTICULATE MATTER FORMED IN FUELS

Fuels used in an Air Force-sponsored test program were found to have decreased in their thermal stability, and the lowered thermal stability manifested itself by the formation of particulates in the fuel. An investigation to identify the fuel particulates and fuel contaminants responsible for particulate formation was conducted.

A study was also conducted to determine the effects of thermally stressing two test fuels, both while they were in contact with one of several metals and in the absence of metals.

Experimental and Results

Particulate Analysis

Two of the specimens examined during the course of this study consisted of particulate material contained on filters. In one case, a 1- to 2-mm spot of material was contained on a Millipore® membrane filter. The second particulate deposit was contained on a paper filter. Both residues were formed during the JFTOT testing of special test fuels. The deposits were found to be insoluble in common organic solvents. Examination of both deposits by the energy dispersive x-ray fluorescence technique showed that they contained no significant quantities of metals. The Millipore filter deposit was washed with a small quantity of methylene chloride, and the resulting rinse was analyzed by gas chromatography/mass spectrometry (GC/MS). The only materials detected were traces of di- and tricyclic aromatics. These compounds are likely due to nonvolatile traces of fuel which remained on the filter.

The bulk of the particulate deposit still remained on the Millipore filter and the spot was not noticeably lightened by the solvent ringe. An infrared attenuated total reflectance (ATR) spectrum was recorded of the filter spot after the previous solvent ringe. Only the spectrum of the filter material, polyvinyl acetate, was obtained. It was concluded that the deposit was carbonaceous and had very little organic character.

Fuel Contaminant Analysis

Several experimental fuel blends which had failed the JFTOT test, with resultant filter plugging, were examined to determine the nature of their contaminants. Two GE/TJ test fuels, coded 8AR-12% and 8XY-13%, were compared to earlier samples of the same fuels that had shown satisfactory thermal stability. Differential spectrometric techniques (GC/MS, IR, and UV absorption) were employed to find compositional differences between the fuels, but no organic contamination was detected in the test fuels by these techniques.

Emission spectrographic analyses of the fuels were conducted to determine any difference in metals content. For semiquantitative results, the fuels were extracted with dilute UltraR metals-free hydrochloric acid. This approach allows metals to be concentrated in the acid layer, a portion of which is then evaporated in the cup of a spectrographic electrode. Most metals are efficiently extracted in this manner. A few, such as silicon and aluminum, are not. Any significant amount of these metals tends to form a scum at the fuel/acid interface. In conducting the analyses, therefore, a specimen taken from the fuel/acid boundary was also analyzed. Analytical results are presented in Table 29.

Trace metals were determined in the same way in two fuels which were presumed to be identical, but which produced significantly different thermal stability test results. The two fuels were

rij.

SPECTROGRAPHIC ANALYSIS OF FUEL EXTRACTS AND EXTRACTION INTERFACES TABLE 29.

Analysis	İ		q	nd Qualit	ative he	PPb and Qualitative Amounts of Matals* Found is Pollandan Pusls	Tetals 1	Powed in	Pollanie	e Feel		
Request No.:		78015	387	9018	¥	78015	78015	15	*	90.00	F	78026
Type sample: JP-6 Fuel		tro1	# \\	Test C2/T3-78-	Š	Control	Test (4)	Test C2/43-79-	# / ·	Thet		There
Designation: GE/TJ-71	GE/TJ-7	8-64R-128	_,	BAR-12.0-06	7-62/20	02/TJ-78-6XY-130	1-120	err-13.0-06	Pomod	Powed Can (A)	Pectang	Sectionaries Can (8)
Hotal	Puel	Surface	192	Surface Fuel Surface		Past Surface Past	7	Surface	Z	Surface fuel	120	Sections
2		trace	110		•	4 trace	•	trace		trace		trace
8	٧٦	trace	•	trace	•	trace	•	trace	•	trace	•	trace
£			•		0.1-1.0	trace	0.1-1.0	trace		trace	0.1-1.0	trace
£	•		21		→		11		2) 	×	
Ē	0.1-1.0	_	*	trace			\$	trace			m	
3			•	minor		ma jor		10 JOE				ménor
78		ma jor		me jor		me jor		## jor		trace		me for
14		trace		ma jor		trace		me jor		trace		trace
2		minor		minor		minor		trace		30 jor		trace
2		trace				trace		trace		trace		trace
5		-	0.1-1.0			trace		trace				-
>						trace				trace		
8											170	
Z		-	0.1-1.0									
,						trace						
I						trace						

"Most metals could be detected at levels as low as 0.2-1.0 ppb, except for zinc which can be detected only as low as 50 ppb. For the qualitative analyses of the fuel/extract interface surface, a "major" designation isdicates that this metal consisted of 10-100% of the total metals detected, a "minor" designation means 1-10% of the total metals, and "trace" means less than 1%.

numbered identically, JP-8-Sple 2-cell 304-1/2, with the only visible difference between the two samples being in the containing cans. The sample called "fuel A" was contained in a round welded steel epoxy-coated can. The other, called "fuel B," was contained in a rectangular soldered tin can. The metals analyses for these fuels are also presented in Table 29.

An examination of the analytical data showed that, for GE/TJ-78 samples 8AR-12 and 8XY-13, the test fuels contained more and significantly higher levels of trace metals than the control fuels. Tin appeared at a moderately high level in both test fuels but not in the controls. Iron was high in one test fuel but not in the control. The presence of a high level of zinc (170 ppb) and increased levels of copper and lead were the major differences found between fuel A and fuel B. High concentrations of trace metals are, of course, associated with decreased thermal stability in hydrocarbon fuels.

Thermal Stressing Study

Fuels A and B, labelled JP-8-Sple 2-cell 304-1/2, were thermally stressed under conditions that simulated a JFTOT test run. These fuels were heated in 6-inch by 1/4-inch ID glass-lined bombs containing single 1-inch strips of various metals, for periods of 15 and 40 minutes at 300°C. Air was excluded from the test cells, and fuels samples without metal strips were heated as controls. The heated fuels were then filtered dropwise through one spot on a 0.45-µm Millipore filter to isolate any particulate residue. The intensity and size of the residue spot was found to be a-good visual indicator of the amount of particulate formed. Residue spots were thus given relative numerical ratings based on their approximate size and intensity; these are presented in Table 30. A photograph of all filter spots is shown in Figure 46. Emission spectrographic analyses were conducted on the residues from the 40-minute heating period to determine the presence of the

TABLE 30. ANALYSIS OF RESIDUES FORMED BY HEATING FUELS AT 300°C IN CONTACT WITH SELECTED METALS

Fuel Container	Contact Metal	Time at 300°C, min	Residue Intensity Rating [®]	Metals Detected ^b in Filtered Residues				
				Al	Cu	Pb	Sn	Zn
Round	none	nc 😘	0	slt	no	no	no	no
(fuel A)	none) #	2					
	none	40	2	yes	slt	no	no	no
	Al	15	2	_				
	Al	40	3	yes				
	Cu	15	3 1	•				
	Cu	40	3		yes			
	Pb	15	3		•			
	Pb	40	2			yes		
	Sn	15	2			-		
	Sn	40	1				no	
	Zn	15	1					
	Zn	40	0					no
Rectangular	none	none	0	slt	no	no	no	no
(fuel B)	none	15	2					
	none	40	3	yes	slt	no	no	no
	Al.	15	2	-				
	A1	40	2	yes				
	Cu	15	1	_				1 , .
	Cu	40	3		yes.			
	Pb	15	3		-			
	Pb	40	3			yes		
	Sıı	15	2			-		
	Sn	40	1				no	
	Zn	15	3					
	Zn	40	3					no

Refers to relative quantity of residue present on filter: 0 = none apparent; 1 = gray spot about 1/8 in. diameter; 2 = plack spot about 1/8 in. diameter; 3 = black spot about 1/4 in. diameter.

The following approximate quantities of metals can be detected by emission spectroscopy: 0.2 µg for aluminum and copper, 1 µg for tin and lead, and 50 µg for zinc. A "no" designation means none was detected, "slt" means a slight indication of the metal near the minimum detectability level, and "yes" means a strong indication of the metal at an undetermined concentration.

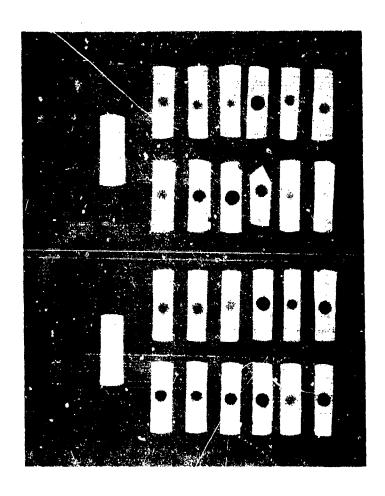


Figure 46. Photograph of residues collected on membrane filters from fuels heated at 300°C for 15- and 40-minute periods.

individual contact metals in the particulate matter. These data are included in Table 30. Aluminum, copper, and lead were detected in residues obtained from both fuel A and fuel B after heating them in the presence of the respective metals. Aluminum and copper, however, were found in the particulates after heating the fuels without contact metals.

Discussion and Conclusions

The thermal stressing studies showed some unexpected results. In several cases, with tin as the contact metal for example, the amount of particulate matter formed in 40 minutes appears to be less than that formed in 15 minutes. The particulate material formed in the fuel is extremely difficult to remove from a glass vessel surface after it has settled. The static nature of the test may have allowed greater settling of the material over the longer period of time. Thus the low observed amounts may represent poor recovery of the particulate rather than a lower rate of formation.

The two JP-8 fuels, A and B, reportedly give greatly differing results in JFTOT tests. Though not consistent for all metals, the particulate residue appeared to be generally heavier in fuel B. The difference found in trace metals content was sufficient to account for the poor thermal stability in JP-8, B.

A high trace metals content similarly appeared to be the likely cause of JFTOT failure by the modified JP-8 (8AR-12 and 8XY-13) fuels.

10. PCB ANALYSIS OF UNKNOWN WASTE FLUID

Several drums of a waste fluid material having unknown identification, composition, and history were received at AFAPL.

Analytical data were required on its PCB content, if any, in

order to facilitate proper disposal of the material. A single sample was submitted for analysis to determine approximate PCB concentration.

Analytical Procedure

The oil-like fluid was diluted 1:9 with hexane and then analyzed on an HP5830A gas chromatograph fitted with an electron capture detector:

Column - 1% SP 2250 on 100/120 mesh Supelcoport in 6 foot x 1/4 inch glass

Col. Temperature - 190°C

Carrier Gas - 95% argon/5% methane at 30 ml/minute

The sample chromatogram was compared with those for standard Aroclors 1242 and 1254 which had also been diluted 1:9 with hexane prior to analysis.

Results

A trace (0.91 ppm) of PCB was detected in the sample labeled A-79008. This is much below the current EPA action level of 500 ppm. These results were reported by telephone to the request initiator.

11. ANALYSIS OF DEPOSITS ON JFTOT PREFILTERS

Deposits were formed on the prefilters (0.45-µm porosity Millipore) for the Jet Fuel Thermal Oxidation Tester during the testing of two diesel fuels. Two prefilter samples submitted for analysis of deposits were labeled:

Sample 1 - prefilter diesel fuel #2, Run 1, J79, 12/4/79

Sample 2 - prefilter diesel fuel #2, Run 1, J79, 12/6/79

Chemical analyses were conducted to determine the nature of these deposits. Both iron oxide and organic deposits were suspected. A control filter with residue and a filter blank were also analyzed to aid in the interpretation of results.

Emission Spectrographic Analyses

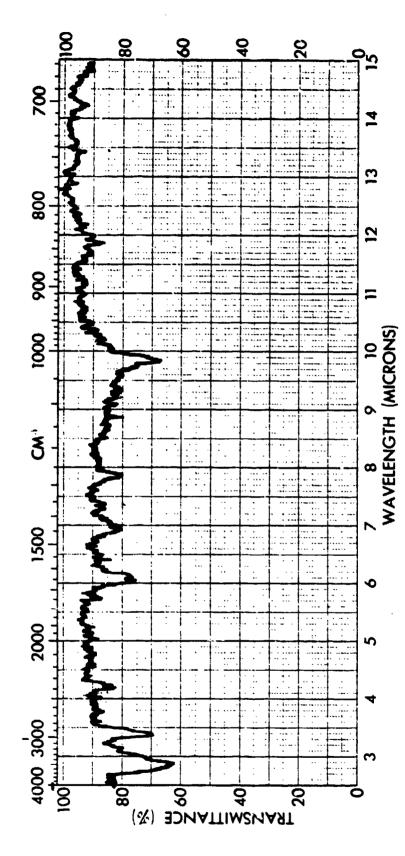
Deposits from the two sample prefilters and the control filter (labeled clean DF-2) were subjected to emission spectrographic analysis. Prior to analysis, the deposits were removed from the filters with a spatula using gentle abrasion in the presence of lithium carbonate. The latter material was used as a matrix diluent for analysis. The analytical results showed a number of elements to be common to both the control and sample filter deposits at nearly the same levels. These elements included major amounts of iron, aluminum, and sodium; moderate amounts of silicon, tin, copper, and lead; and small amounts of magnesium, manganese, and titanium. The only significant difference between the control and sample deposits were: (a) run 1 sample contained a major amount of nickel and (b) both run 1 and run 2 samples contained trace amounts of silver.

Infrared (IR) Spectrographic Analyses

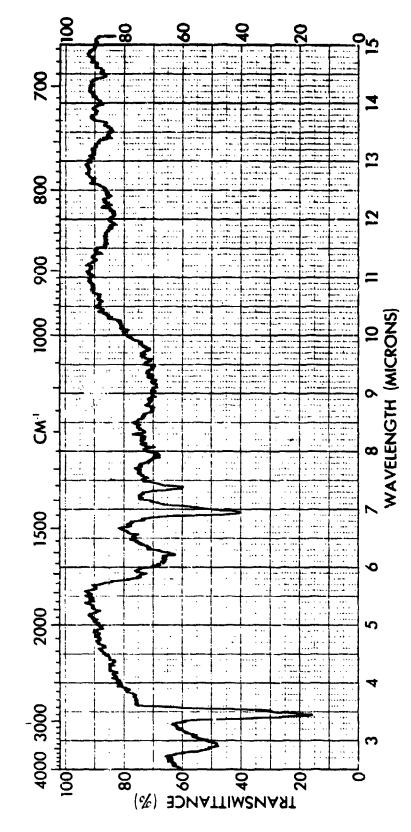
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Deposits were removed from the sample and control filters by gently rubbing a spatula across the filter surfaces in the presence of potassium bromide (KBr). The KBr/deposit mixtures were then compressed into pellets for standard IR analyses. A piece of the blank filter disk was analyzed in order to determine major absorption bands characteristic of the filter materials.

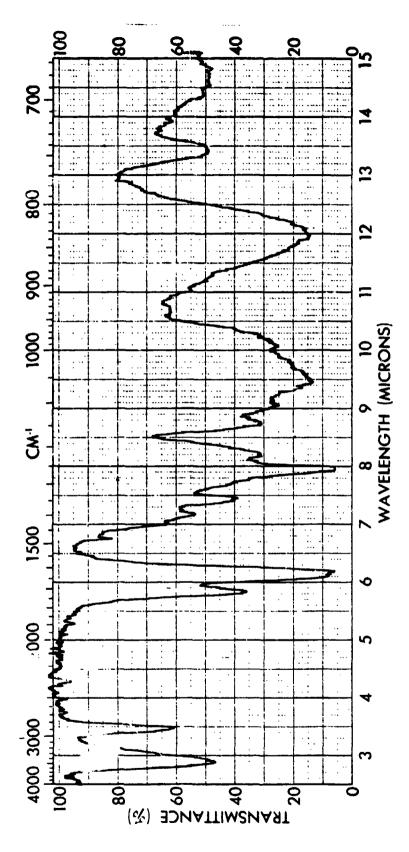
Infrared spectra for sample run 1, the control, and the blank filter are shown in Figures 47-49. The spectrum for sample run 2 was identical to that of run 1 and is not included with the



IR spectrum for prefilter deposit from sample run #1



IR spectrum for prefilter deposit from control run with clean DF-2. Figure 48.



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Figure 49. IR spectrum of Millipore filter.

figures. The spectrum of the Millipore filter indicates that it was a mixture of cellulose nitrate and acetate. Spectra for the control and both sample deposits show the presence of cellulose from the Millipore filter, and aliphatic hydrocarbons which were probably diesel fuel components. An additional absorption band at approximately 9.85 microns wavelength is observed for the sample deposits but not for the control deposit. This band is usually characteristic of low molecular weight oxides, although sulfates also show a similar band. The amount of sample available for analysis was insufficient for precise identification.

The absence of significant absorption bands not found in the control indicates that the sample deposits were largely inorganic in nature. Based on the emission spectrographic results and the limited infrared spectral data, the deposit appears to have consisted largely of metal oxides.

12. HPLC ANALYSIS OF MIL-L-7808 FLUID SAMPLES FOR OIL ADDITIVES

Five Air Force fluids, coded TEL-0032 through TEL-0036, were analyzed for oil additives by a high pressure liquid chromatography (HPLC) method developed on a previous Monsanto Research Corporation contract. This method is described in Report AFAPL-TR-78-50, "Reclamation of Synthetic Turbine Engine Oil Mixtures." The analyses were required because the Air Force had identified a production batch of MIL-L-7808 fluid which was suspect with respect to the concentration of additives in the oil.

Analysis Procedure

The HPLC analyses were performed using a Waters instrument having a Model 660 Solvent Programmer, an ultraviolet detector and an HP-3380A integrator. The following analytical conditions were utilized:

Column: Partisil PX5 10/25 PAC

Sample Concentration: 10 µl of 10% sample in cyclohexane

Elution Solvent: Programmed from isooctane to 30/70 iso-

octane/methylene chloride over a 20

minute period

Flow Rate: 2 ml/minute

Detector Wavelength: 254 nm

Results and Discussion

The five MIL-L-7808 fluids were analyzed for five oil additives having the elution times shown in Table 31. The analytical results, shown as weight percents in Table 32, were calculated using peak area values obtained from the integrator for both the samples and standards. Individual additive standards were prepared in either cyclohexane or methylene chloride. A sample density value of 0.94 g/ml was used for all calculations. A typical sample chromatogram is shown in Figure 50.

Since the elution times for PANA and DOPTA were very close, there was some initial concern that the indicated PANA peaks in the samples may actually be a combination of both additives. However, the analysis of a mixed standard showed that the Partisil column would resolve the two additives if they were both present in a sample. The elution times for the single component in all samples in the 11-13 minute range matched well with that for PANA.

TABLE 31. HPLC ELUTION TIMES FOR MIL-L-7808G OIL ADDITIVES

Oil additive	Abbreviation	Minutes
4,4'-Dioctyldiphenylamine	DODPA	9.62
3,7-Dioctylphenothiazine	DOPTA	11.84
N-phenyl-α-naphthylamine	PANA	12.01
Phenothiazine	PTA	15.31
Tricresyl phosphate	TCP	23.59, 24.06,
•		24.32, 25.70

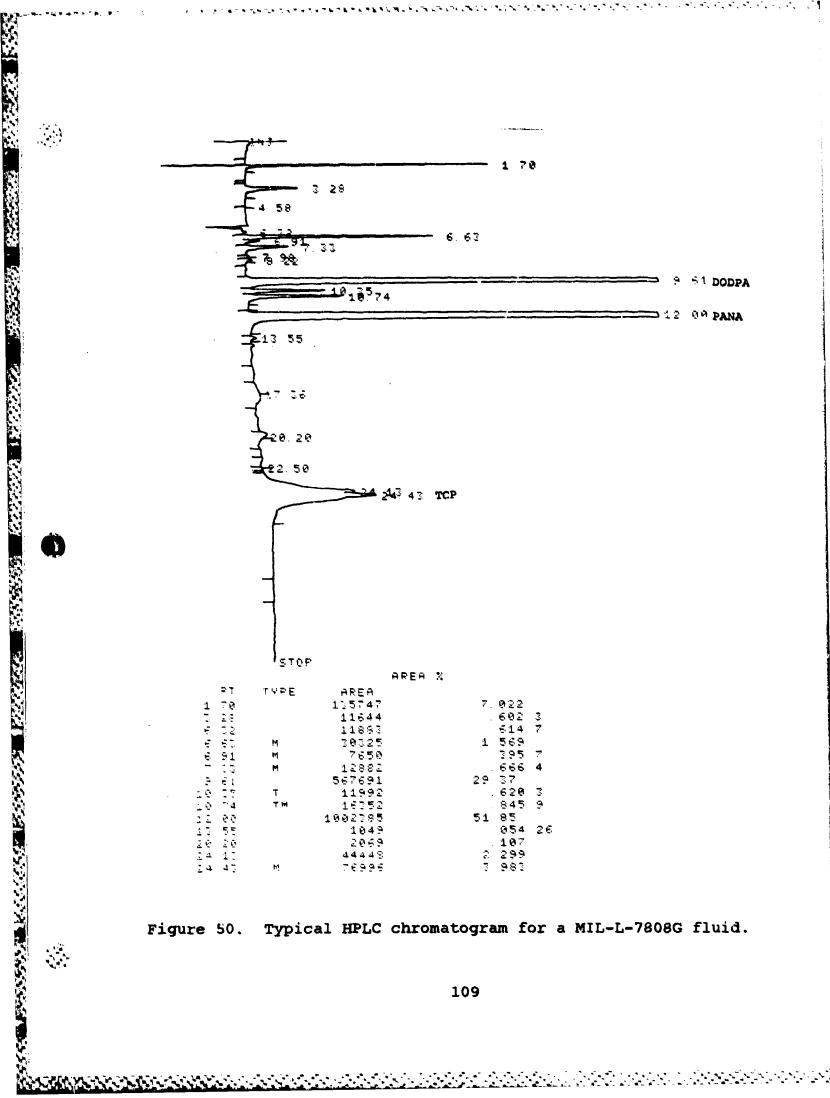


TABLE 32. ADDITIVE CONCENTRATIONS IN MIL-L-7808G FLUIDS

	Weigh	nt perce	nts of	additi	ves
Fluid	DODPA	DOPTA	PANA	PTA	TCP
TEL-0032	1.14	ND a	0.43	ND	2.17
TEL-0033	1.17	ND	0.83	ND	0.55
TEL-0034	1.14	ND	0.43	ND	2.41
TEL-0035	1.10	ND	0.43	ND	2.43
TEL-0036	1.15	ND	0.85	ND	0.36

and - not detected at the low ppm levels.

13. PERFORMANCE OF COMBUSTIBLE GAS MONITORS IN DETECTING JP-9, JP-10, AND RJ-6 VAPORS

Fuel vapor monitors currently in use by the Air Force were designed and calibrated primarily for the detection of JP-4 vapors in air. The Air Force also has a need to monitor workplace environments in which vapors of high density fuels (JP-9, JP-10, and RJ-6) may be encountered.

Chemical composition, diffusivity, and thermal conductivity are the properties of fuel vapors that have the greatest effect on the response characteristics of fuel vapor monitors commonly used at Air Force facilities. These properties differ for JP-4 and high density fuel vapors. Therefore, the Aero Propulsion Laboratory sought to determine some response characteristics of selected fuel vapor monitors to high density fuel/air mixtures.

The response of the following monitors to high density fuel/air mixtures was investigated in this work:

Combustible Gas Indicator, Model TBA5100-1, from Beckman Instruments, Inc.

Portable Combustible Gas Alarm, Model 100S, from Mine Safety Appliances Company

Hydrocarbon Super Surveyor, Model 1314 (GasTechtor), from GasTech, Inc.

Two units of the Beckman instrument were included in this investigation. The instrument from GasTech, Inc., was included as a reference monitor since a significant data base had been developed for its response to mixtures of low vapor pressure jet fuels with air (ref. 4). A flame ionization detector, in a Varian Model 1440 chromatographic analyzer, served as a reference sensor for hydrocarbon vapors.

Experimental

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The JP-9, JP-10, and RJ-6 fuel samples used in this investigation were supplied by the Fuels Branch of the Aero Propulsion Laboratory. They carried the following identifications:

JP-9: drawn on 12 May 1980 from a 55-gallon drum, Batch 39.

JP-10: drawn on 8 April 1980 from a 55-gallon drum (No. 31) manufactured in November 1979 by Ashland Chemical Company; Batch 9317, 9335-01-048-5295.

RJ-6: drawn on 14 May 1980 from a 55-gallon drum (No. 11) manufactured in October 1979 by Ashland Chemical Company, Batch 9289.

The apparatus used in this work was designed and built with the following objectives:

- To generate hydrocarbon/air mixtures, from fuel having different volatilities, in a continuous manner in the laboratory;
- 2. To monitor hydrocarbon concentrations with the instruments whose performance was to be characterized; and
- 3. To determine hydrocarbon concentrations simultaneously with the reference instrument.

The system used for performance characterization of combustible gas monitors is shown in Figures 51 and 52. The hydrocarbon vapor/air mixtures were generated by bubbling air at a predetermined rate through liquid hydrocarbon contained in the sparger. The hydrocarbon content of the vapor/air mixture was regulated by maintaining a desired sparger temperature with a surrounding constant temperature bath, by controlling the air flow rate through the sparger, and also by diluting the fuel/air mixture with a diluent air stream.

The hydrocarbon/air blend was thoroughly mixed. Samples were drawn into the instruments through probes that extended into the sampling manifold.

A Varian Model 1440 chromatographic analyzer incorporating a flame ionization detector (FID) was used as the reference instrument. This type of detector was selected because its response is known to be nearly linearly related to carbon (in the form of $\mathrm{CH_{n}}$) concentration in gaseous samples (ref. 5). The lower explosive limit (LEL) of aliphatic hydrocarbon vapors of different compositions can also be expressed as a function of carbon (or $\mathrm{CH_{n}}$) content (~75,000 ppm). Zabetakis has reported that the vapors of JP-4 and similar fuels contain approximately 48 mg/l of the fuel in air at the lower flammability limit (ref. 6).

Monitor responses, including those of the flame ionization detector, were recorded at the 1-minute time point after stable gas flow conditions had been established, and at 2-minute intervals thereafter. Under each set of selected flow conditions, monitor responses were recorded for at least a 10-minute period. For quantified comparison of response data, the concentrations indicated by the instruments at the 5-minute time points were used. As indicated in Figure 53, no significant hydrocarbon vapor

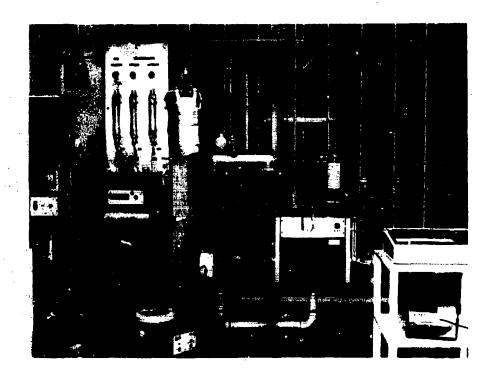


Figure 51. Apparatus for generating and monitoring fuel vapor/air mixtures.

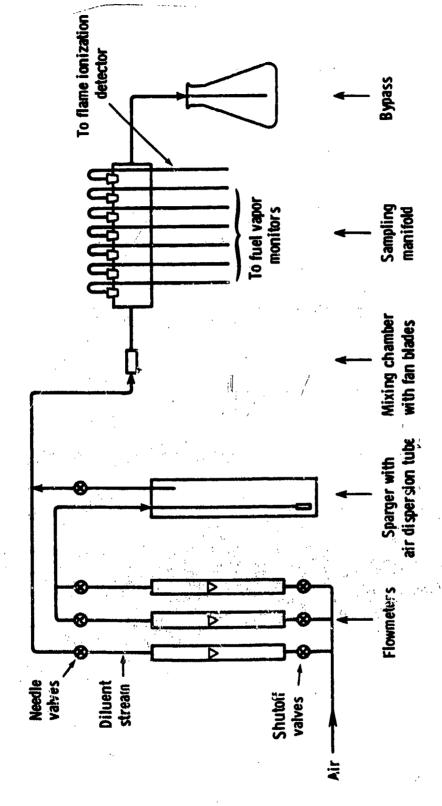
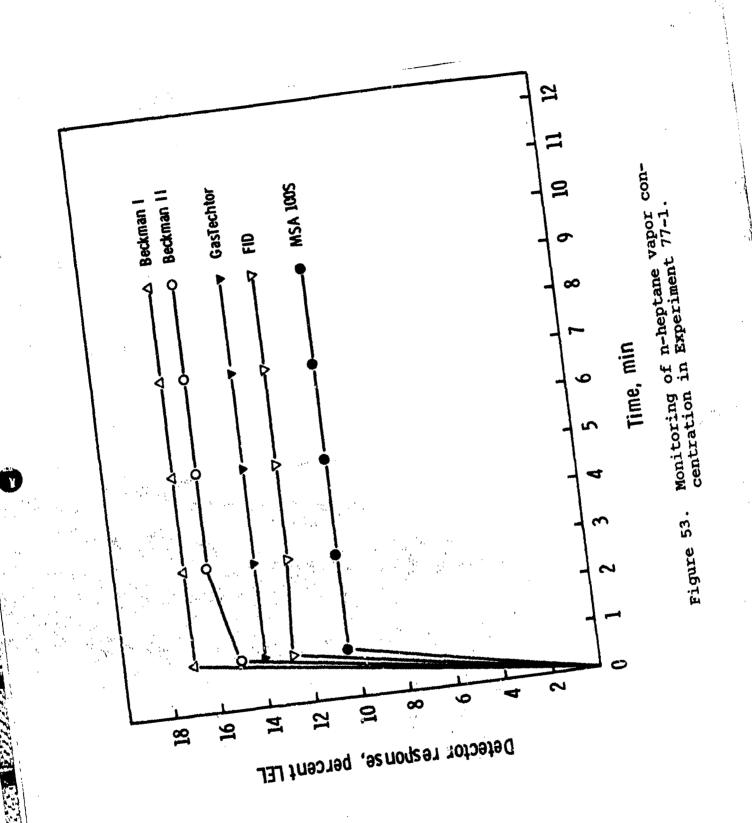


Figure 52. Schematic of apparatus used to generate and monitor fuel vapors.



concentration change was observable after a 5-minute flow of the vapor/air mixture through the system, when heptane was used as the combustible hydrocarbon.

Additional details regarding the apparatus, and its calibration and operation, may be found in a recent report on performance characterization of combustible gas monitors in detecting jet fuel vapors (ref. 4).

Results and Discussion

Figure 54 depicts combustible gas monitor responses in the course of an experiment on JP-9 vapor detection. If fuels contain components of different volatilities, lowering of fuel concentration with time is observed if the temperature of the fuel remains constant.

n-Heptane was included among the combustible materials tested in this investigation because it is a frequently used calibration substance for the monitors, and its vapor pressure and concentration remain constant during an experiment.

The results of the individual experiments with the four combustible substances are listed in Tables 33 through 36. The results for each experiment are listed in the order of diminishing monitor response intensity. The conditions used during the experiments are shown in Table 37.

To facilitate data analysis, the response values of each instrument in sensing a specific combustible gas were averaged. Thus, a single number characterizing the performance of each instrument in sensing each gas was obtained (see Table 38). Normalized responses of instruments with respect to the concentration determined with the flame ionization detector were also calculated. These are presented in Table 39 and Figure 55.

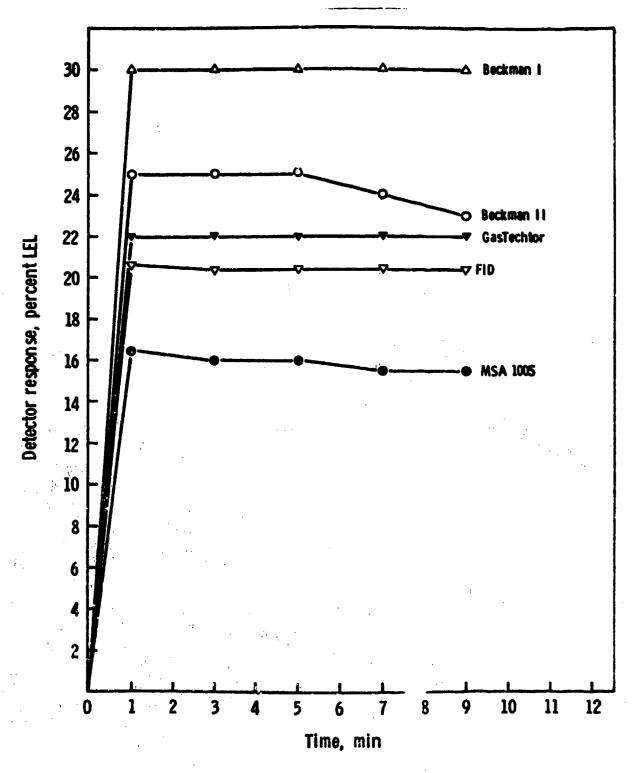


Figure 54. Monitoring of JP-9 vapor concentration in Experiment 81-2.

TABLE 33. MONITURING OF n-HEPTANE VAPOR CONCENTRATIONS, DETECTOR RESPONSES AT 5-MINUTE TIME POINTS

2	60 58 46	42.5	34.5
Exp. 77-2	Beckman I 60 Beckman II 58 Gastechtor 46	FID	MSA 100S
	17 16 14	12.6	10.5
Exp. 77-1	Beckman I 17 Beckman II 16 GasTechtor 14	FID	MSA 100S
2	60 58 46	41.1	
Exp. 74-2	Beckman I 60 Beckman II 58 GasTechtor 46	FID	
	18 16 14	11.7	
Exp. 74-1	Beckman I 18 Beckman II 16 GasTechtor 14	FID	

MONITORING OF JP-9 VAPOR CONCENTRATIONS, DETECTOR RESPONSES AT 5-MINUTE TIME POINTS TABLE 34.

	31 T 8	6.9	6.5
Eup. 63-3	Beckman I 16 Beckman II 11 Casfechtor 8	13.6 FID	MSA 1008
		13.6	12
Exp. 83-2	Beckman I 23 Beckman II 20 GasTechtor 15	32.3 FID	24.5 MSA 100S
-	45 38	32.3	24.5
Esp. 63-1	Beckman I 45 Beckman II 38 GasTechtor 33	F 10	17 MSA 100S
7	30 23	21.2 FID	1.
Exp. 82-2	Backman I 30 Beckman II 28 GasTechtor 23	PID	41 MSA 100S 31.5
1	50	41.1 FID	31.5
Exp. 82-1	Beckman I 50 Beckman II 48	FID	GasTechtor MSA 100S
7	30 23 23	20.4	16
Exp. 81-1 Exp. 81-2	Beckman I 30 Beckman II 25 GasTechtor 23	FID	MSA 1308
-	50 £	38.3 FID	38
Exp. 81-	Beckman I 50 Beckman II 43	FID	GasTechtor 38 MSA 100S MSA 100S 28

MONITORING OF JP-10 VAPOR CONCENTRATIONS, DETECTOR RESPONSES AT 10-MINUTE TIME POINTS TABLE 35.

6-3	2 H 6 H 7 H 7 H 7 H 7 H 7 H 7 H 7 H 7 H 7		
Ä	Pochess I 6 Pochess II 3 Castocktor 2		
7	# # # # # # # # # # # # # # # # # # #		
E. 6	Beckman I 7 Beckman I 6 Beckman II 5 Beckman II 3 Garfredtor 4 Cantechtor 2 Han 100e 2	 	
-	9 •	3	• in
Exp. 90-	Beckman I 10 Gasfachtor B	3.1 710	HEA 1006 Beckmen 11
e e	H	3.1	
Esp. 79-	Beckman I 11 Beckman II 6 GasTechtor 4 1083 1005 3.5 (£	
2	15 7 6.5	6.0 FTB	
3-3 Etq. 79-1 Exq. 79-2 Exq. 79-3 Exq. 60-1 Exq. 60-2 Exq. 60-3	Becken I 15 GasTechtor 8 Becken II 7 MSA 1005 6.5	92	
<u> </u>	20 23	14.8 PID	2 a
E. 79	Beckman I 23 Gasfechtor 20	12.6 FID	MEA 1005 14.5 Beckman II 11
5	25 r 15 t 13	12.6	
Eup. 73-1 Eup. 73-2 Eup. 73-	Beckman I 25 Garffechtor 15 Beckman II 13	5.6 P.ID	
7	15 1 10 1 7	5.6	
Erp. 73	Beckman I 15 Beckman II 10 GasTechtor 7	2.9 F1D	
1-62	1 6 11 6 3r 4	2.9	
H	Beckman I 8 Beckman II 6 Sasfechtor 4	9	

MONITORING OF RJ-6 VAPOR CONCENTRATIONS, DETECTOR RESPONSES AT 5-MINUTE TIME POINTS TABLE 36.

ī	~	, ,	
Pro-	MEA 1005	1 4 PTD	Beckman I Beckman I
3	. m.		00
P. 0	MEA 1008	3.0 Pro	Section I Section I
7	4.5	3.0	
Exp. 86	MSA 1008 GesTechton	rio	Beckman I Beckman II
5	 	1.7 7.10	ä
Esp. 85	Gesfechtor MGA 1005 Beckman 1	FID	Beckman II
9	n 4 4	3.3 FID	m .
Esp. 85	Beckman I GasTechto	7.6 FID	Beckman II
7	리 2 명 8 · S	7.6	w
. 84-3 No. 85-1 Lo. 85-2 No. 85-3 Lo. 86-1 Lo. 86-2 No. 86-3	GasTechtor 8 GasTechtor 4 MSA 1005 2.5 GasTechtor 11 Beckman I 5 GasTechtor 3 MSA 1605 7.5 MSA 1008 4 GasTechtor 2 MSA 1005 4 Beckman I 2 GasTechtor 4 GasTechtor 2 MSA 1005 3 MSA 1005 2	710	Beckman I 3 Beckman II 2 Beckman II 1 Beckman II 5 Beckman II 3 Beckman I 1 Beckman I 0 Beckman I 0 Beckman I 0 Beckman I 0 Beckman I 0 Beckman I 1 Beckman I 1 Beckman II 0 Beckman II 0
ات	2 2	1.8 PID	-1 O
END. 84	MSA 1005 Gestechtor	3.2 FID	Beckman II Beckman I
7	+ +	3.2	70
Erg. 84-1 Erg. 84-2 Erg.	GasTechto MSA 1008	7.0 PID	Beckman I
7-1	r 8	7.0	1 3
Exp.	GasTechto MSA 160s	9	Beckman I Beckman I

TABLE 37. EXPERIMENTAL CONDITIONS DURING PERFORMANCE CHARACTERIZATION OF COMBUSTIBLE GAS MONITORS

HC/fuela	Experiment number	T,b	Ra, 1/min	Rda, 1/min
n-Heptane	74-1	0	1.0	11.0
	74-2	0	3.9	8.1
	75-1	0	1.0	11.0
	75-2	0	3.9	8.1
	76-1	0	1.0	11.0
	76-2	0	3.9	8.1
	77-1	0	1.0	11.0
	77-2	0	3.9	8.1
JP-9	81-1	21.1	3.0	3.0
	81-2	21.1	1.5	4.5
	82-1	21.1	3.0	3.0
	82-2	21.1	1.5	4.5
	83-1	0	6.0	-
	83-2	0	3.0	3.0
	83-3	0	1.5	4.5
JP-10	73-1	21.1	3.0	9.0
	73-2	21.1	6.0	6.0
	73-3	21.1	6.0	_
	79-1	21.1	6.0	- .
	79-2	21.1	3.0	3.0
	79-3	21.1	1.5	4.5
	80-1	0	6.0	-
	80-2	0	3.0	3.0
	80-3	0	1.5	4.5
RJ-6	84-1	21.1	6.0	•
	84-2	21.1	3.0	3.0
	84-3	21.1	1.5	4.5
	85-1	21.1	6.0	-
	85-2	21.1	3.0	3.0
	85-3	21.1	1.5	4.5
	86-1	0	6.0	_
	86-2	0	3.0	3.0
	86-3	0	1.5	4.5

^aHC/fuel - single hydrocarbon or fuel.

bTemperature of the single hydrocarbon or fuel in the sparger.

^CFlow rate of air through liquid hydrocarbon in sparger.

dFlow rate of air in diluent stream.

TABLE 38. SUMMARY OF AVERAGED DETECTOR RESPONSES

Instrument	n-Heptane	JP-9	JP-10	RJ-6
Beckman I	38.7	34.9	13.3	2.4
Beckman II	37.0	30.4	7.3	1.8
GasTechtor	33.0 ^{&}	25.7	8.0	4.2
MSA 100S	22.5	19.4	5.7	4.3
FID	27.0	24.8	6.0	3.3

The averaged value of experimentally determined detector responses for the GasTechtor monitor (30% LEL) was multiplied by 1.11 to correct for calibration of this instrument with hexane.

TABLE 39. SUMMARY OF NORMALIZED DETECTOR RESPONSES

Instrument	n-Heptane	JP-9	JP-10_	RJ-6
Beckman I	143	141	221	74
Beckman II	137	123	122	54
GasTechtor	122	104	133	127
FID	100	100	100	100
MSA 100S	83	78	96	130

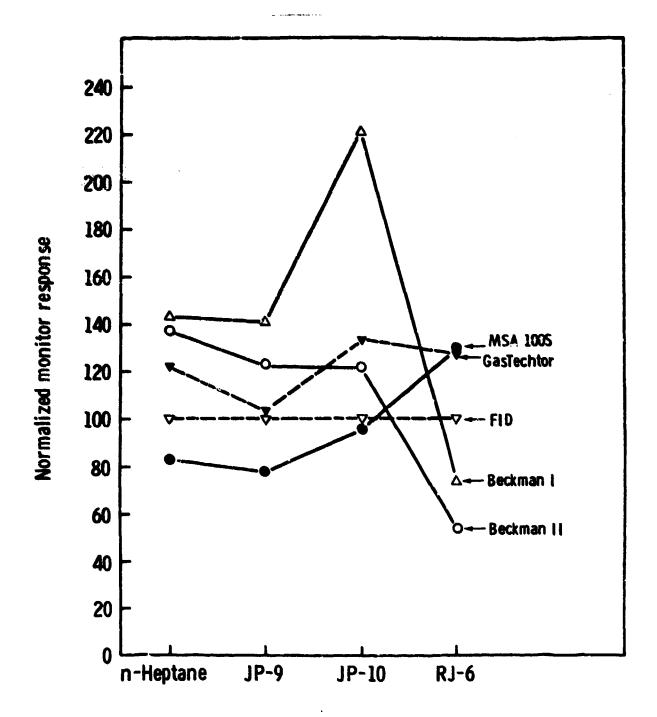


Figure 55. Combustible gas monitor responses normalized to the flame ionization detector response.

All instruments detected combustible vapors emanating from JP-9, JP-10, and RJ-6 in undiluted fuel vapor/air mixtures even when the fuels in the sparger were maintained at 0°C (32°F). Upon three-to-one dilution with air, vapors of JP-9 and JP-10 were still detected. However, the vapors of the lowest vapor pressure fuel included in this investigation, RJ-6, were not always detected upon dilution of the initially equilibrated vapor/air mixture.

The large differences in measured combustible gas concentrations, as determined with the different instruments, were attributed mainly to electronic drifts. Large drifts of the meter zero reading had been experienced in earlier work with short warm-up time (ref. 4). Upon that observation, a minimum warm-up time of 0.5 hr was used in all experiments. Despite this precaution, zero drifts ranging up to 10% LEL were experienced with the Beckman I monitor in the course of a sequence of experiments. At low combustible vapor concentration, such an error has a large effect on the value of the measured concentration, and also on the normalized response (see Table 39 and Figure 55).

Conclusions

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- 1. The response characteristics of instruments included in this investigation were such that, despite a lack of accuracy, they would provide adequate warning when the workplace atmosphere becomes contaminated with JP-9, JP-10, or RJ-6 vapors. (This conclusion was based upon the presumption that the monitor alarms are set to provide audible or visible warning signals when the flammable vapor concentration in air reaches 25% of the LEL value.)
- 2. The combustible gas monitors did not enable accurate flammable vapor concentration measurements in air (see ref. 7).

- 3. The differences in results obtained with instruments produced by the same manufacturer, and carrying identical model designations, can be great.
- 4. Electronic instability was a major reason for the inaccuracy of concentration values determined with the Combustible Gas Indicator, Model TBA5100-1, manufactured by Beckman Instruments, Inc.
- 5. The concentrations of combustible vapors above JP-9, JP-10, and RJ-6 were 41%, 15%, and 8% LEL, respectively, when these fuels were equilibrated with air at 21°C (70°F).

Recommendations

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- 1. Combustible gas monitors should be turned on and operated in standby mode for at least half an hour before use to stabilize the functioning of their electronic components.
- 2. For operation at temperatures above 32°C (90°F), an octane-air mixture is recommended as the calibration gas. A pre-mixed supply is most suitable for monitors that use small amounts of calibration gas, such as those included in this investigation.

14. ANALYSIS OF DEPOSITS ON A-10 AIRCRAFT AND SUPPLY TRUCK FUEL FILTERS

An investigation was conducted to determine the cause of fuel filter plugging in an A-10 aircraft. The investigation involved a detailed examination of the subject filter as well as the filter from a fuel supply truck. The techniques used for analysis were infrared spectrophotometry, emission spectroscopy and light microscopy.

Procedure and Results

The plastic ends were cut away from the A-10 and supply truck filters and sections of each filter were removed. The sections were then cut into small pieces and extracted with methylene chloride. The solvent from each extraction was carefully reduced in volume to a fraction of a milliliter. These small amounts of extract were then placed on rock salt plates and allowed to completely evaporate. Infrared absorption spectra of the residues showed weak bands characteristic of hydrocarbons. From the obviously small amount of material giving rise to the bands, and the nature of the overall absorption spectrum, it was concluded that the hydrocarbons were traces of jet fuel. Approximately the same amount was obtained from each filter.

The supply truck filter contained no residue and was generally very clean. The aircraft filter, however, had relatively large amounts of readily removable residue which was light and partially fibrous in nature. An emission spectrographic analysis of this residue was conducted to determine its metals content. Results are presented in Table 40.

TABLE 40. EMISSION SPECTROGRAPHIC ANALYSIS OF RESIDUE FROM A-10 AIRCRAFT FUEL FILTER

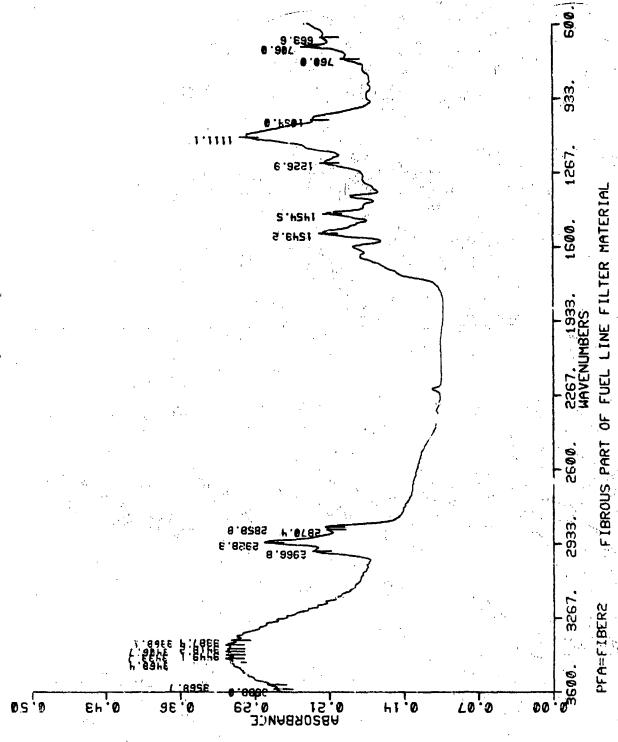
Element	Weight percent
Silicon	5
Calcium	3
Iron	2
Aluminum	2
Magnesium	0.8
Silver	0.7
Cadmium	0.6
Copper	0.6
Chromium	0.4
Tin	0.3
Sodium	0.3
Titanium	0.2
Manganese	0.1
Nickel	0.08

A portion of the A-10 filter residue was ground with potassium bromide (KBr) which was then used to press a pellet for infrared absorption analysis. Due to the nature of the material, the KBr pellet lacked the necessary transparency for a satisfactory spectrum to be obtained. Another KBr/sample pellet was prepared for analysis using Fourire Transform Infrared (FTIR) Spectroscopy. For this purpose, a Digilab FTS-15B Spectrometer was used. The general infrared pattern obtained and shown in Figure 56 is characteristic of a polyurethane prepared from a polyalkoxy alcohol. Absorption bands observed in the region (1730-1530 cm⁻¹) are characteristic of Amide I and II functional groups for polyurethanes. The strong broad band at 1111 cm⁻¹ results from the polyether (aliphatic) moiety.

The IR pattern is not a simple, clean spectrum and may contain spectral data for polyurethane degradation products or for poorly formed polymer. The general region (1730-1300 cm⁻¹) contains many weak and overlapping bands which can be related to other amide type groupings. The unidentified, weak absorption at ca 2250-2260 cm⁻¹ may have been due to nitrile (possibly isocyanate) residues.

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The residue obtained from the aircraft fuel filter was obviously not a homogeneous material. It was approximately 70-80% consumed when ignited in a flame. The non-combustible part of the residue doubtless contained some terrestrial dust as suggested by the first five elements in Table 40. The major part of the residue was organic and a major part of the organic material appeared to have originated from the reticulated polyurethane foam used in fuel tanks for fire suppression. Polyurethanes of either the polyester or polyether type are used for that purpose, though the IR data indicated that the polyether type was used in the A-10 aircraft.



Fourier transform infrared spectrum of material from A-10 fuel-line filter. RES=8 DP Figure 55. LEVEL = 6.20

Polyurethanes are known to be attacked by high humidity or water in the bottom of the fuel tank. Additionally, it has been shown that lead and/or tin at the ppm level in the anti-icing additive, ethylene glycol monomethyl ether (EGME), can cause foam degradation (ref. 8). Particles of degraded foam can be readily dislocated from the bulk block and become suspended in the fuel.

A portion of the A-10 fuel filter residue was further examined using light microscopy. By this means, the non-homogeniety of the residue became quite obvious. One puzzling feature of the residue was the presence of some fibers which did not appear to be polyurethane foam related. These appeared to be of a textile nature and in white, red, and blue colors. The IR data does not show the presence of cellulose (cotton), though for low levels of cellulose the major band could be obscured by the multitude of other overlapping bands. It could not be determined from the IR data whether the fibers were nylon because the amide bands from the polyurethane would obscure the polyamide (nylon) bands in the spectrum.

Conclusions

The residue which appeared on the A-10 aircraft filter did not appear on the filter from the supply truck and there was no evidence to suggest that the residue originated from that source. Rather, the residue appeared to be a collection of material consisting of dust (dirt) particles, degradation products from fuel tank foam, and fibers with a color and twist which suggested they were of a textile origin. The composition of the textile-type fibers was not established. They could have originated from clothes, or similar sources, but not from the filter itself since the colors did not match that of the filter.

15. MODIFICATION AND EVALUATION OF BALL-ON-CYLINDER FUEL LUBRICITY TESTER

Scope of Work to be Performed

The ball-on-cylinder (BOC) tester is the property of the Aero-propulsion Laboratory at Wright-Patterson Air Force Base. MRC was requested to investigate certain test variables as outlined by the Task Force Leader on the BOC Machine Operations' Task Force Committee. This committee was attempting to improve the precision and significance of the lubricity test. MRC work under this program encompassed three tasks:

- (1) Upgrade the current flow system in the BOC; fill the cracks, crevices, and joints in the BOC test chamber with an epoxy sealant and then smooth the surfaces; and replace the bearings on the shaft rotating the test cylinder.
- (2) Analyze the metallurgy of three test cylinders (numbers 108, 127, and 147) from a previous BOC round robin study to determine significant differences that might explain test results variations.
- (3) Determine the repeatibility of the test method after upgrading the BOC tester.

Experimental

BOC Upgrading

The cracks, crevices, and joints in the BOC test chamber were filled with "EPOXY-PATCH" from Hysol Division of Dexter Corporation. This adhesive had been tested for fuel compatibility by bonding two pieces of glass together and then immersing the bond in JP-4 fuel for 72 hours. No discoloration of the fuel or

softening of the epoxy was observed. Additionally, new bearings for the shaft rotating the test cylinder were purchased and then installed to eliminate a chattering noise and excessive wear.

The BOC flow system was upgraded by recalibrating the flow meter for the wet and dry air inputs used to control relative humidity. The flow meters were calibrated with a Model 63115 Precision Scientific wet test meter having the range of 68-680 liters per hour flow rate. The flow rates at various rotameter settings were determined from the following equation:

$$V_{s} = \frac{P_{m}}{P_{s}} \times \frac{T_{s}}{T_{m}} \times V_{m}$$

where V_s = flow rate in liters per minute, corrected to standard atmospheric conditions

P = barometric pressure, mm Hg, corrected for water vapor pressure at temperature inside wet test meter

 P_{c} = the standard barometric pressure, 760 mm

T_s = the standard temperature in degrees Kelvin, 273.16°K

 T_m = the temperature measured inside the wet test meter, $^{\circ}$ K

V = measured gas flow in liters per minute, determined from the time it takes for a 3-liter volume of air to move through the wet test meter at ambient atmospheric conditions

The flow meter calibrations were conducted over a 3-day period. The barometric pressures, instrument temperatures, and pressure/temperature constants for those days are shown in Table 41.

TABLE 41. PRESSURE AND TEMPERATURE CONSTANTS FOR CALCULATING $\mathbf{V}_{\mathbf{m}}$ ON A PARTICULAR DAY

Date	Instrument temp., °C	Barometric pressure, mm	$\frac{\frac{P_{m}}{P_{s}} \times \frac{T_{s}}{T_{m}}}{\frac{T_{s}}{T_{m}}}$
4/9/80	21.0	736.6	0.8772
4/10/80	23.5	735.9	C.8653
4/11/80	22.5	742.3	0.8775

The calibrated flow rates at various rotameter settings, before and after correction to standard atmospheric conditions, are represented in Table 42 (on the following page) along with the various test data used in the calculations. Significantly, the various values obtained for these rotameter calibrations were considerably different from those which had been used in all preceding tests on this device.

Metallurgy of Tested Cylinders

Test cylinders 108, 127, and 147 from a previous round robin study were tested for surface finish and Rockwell-C hardness. Test results are shown in Table 43.

Cylinder 108 was out of specification on surface finish because there was insufficient unused surface to obtain a proper measurement. Therefore, it was suggested either that future profilometer measurements be made on cylinders before the BOC test or that at least 1/4 inch of unused surface be reserved for subsequent measurements. Cylinder 127 did not meet hardness specifications. Consequently data from this cylinder were invalid.

Cylinders 108 and 127 were then sent to The Dayton Casting Company for chemical and metallography examinations. The chemical composition or both specimens met the specifications of low alloy A1S1 52100 steel. Cylinder 108 had a Brinell hardness of 207 while Cylinder 127 had a Brinell hardness of 217. These readings showed both cylinders to be in the annealed condition. The cylinders were then sectioned and one section each was normalized. in both the annealed and normalized conditions were examined under the microscope at 100 and 500 magnifications, but no defects or processing flaws were detected. The specimens in the annealed condition showed a fine dispersion of carbide in a ferrite matrix; this is normal for the annealed condition. The normalized condition showed that the matrix for both cylinders was comprised of

TABLE 42. FLOWMETER CALIBRATION

Test	Air	Rotameter	Flow Ti	mus for	3-Liter	s* of .	ir in Pet	Flow Times for 3-Liters* of Air in Wet Test Meter	V., 111	liters	V, inters
Date	6	Reading	Minutes	Minutes for Replicate Measurements	plicate	Measure	ments	Average	per mir	Minute	per minute
4-9-80	Drv	0.9	0.9675;	0.9675;0,9675;	0.970;	0.970;	0.970	696.0	3.096		2.716
4-9-80	Drv	7.0	9.775;		0.770; 0.770;			0.772	3.886		3.409
4-9-80	Dry	8.0	0.635; 0.645;		0.645, 0.650;	0.650;	0.645	0.644	4.658		4.086
4-9-80	Drv	0.6	0.560;	0.565;	0.5625;	0.5625;0.565;	0.5623	0.563	5.329	•	4.675
4-9-80	Dry	10.0	0.505;	0.505;	0.505;		0.500	0.504	5.952	6 1	5.221
4-9-80	. Dr.v	11.0	0.455;	0.450;		0.450:		0.450	. 6.567	_	5.848
4-9-80	hry	12.0	0.410;	0.400;				0.404	7.427		6.515
;	;			;	:		;				600
4-9-80	Ket	2.5	5.295;	3.48;	5.73;	2.17;		507.6	CANO	•	597.5
4~9~80	Wet	3.0	3.74;	3.805;	3.78;	3.725;	3.68	3.746	0.13	·.	0.117
4-9-80	Wet	3.5	2.54;	2.47;	2.50;	2.565;	2.535	2.522	0.198	~	0.174
4-9-80	Wet	4.0	1.94;	2.00;	1.955;	1.97,	3.00	1.973	0.,253	_	0.222
4-9-80	Wet	4.5	1.545;	1.545;	1.495;	1.51;	1.52	1.523	0.328	•	0.288
4-9-80	Wet	2.0	1.25;	1.17;	1.25;	1.25;	1.16	1.216	0.410	_	0.360
4-9-80	Wet	5.5	1.065,	0.99;	1.05;	1.06;	0.99	1.031	0.484	•••	0.425
4-9-80	We.	6.0		0.91;	0.91;	0.92;	6.91	0.913	0.54	٠,	0.480
4-9-80	Wet	6.5	0.81;	0.815;	0.81;	0.815;	0.81	0.812	0.613	·	0.538
4-10-80	Wet	7.0	4.54;	4.56;	4.51			4.537	0.661	_	0.572
4-10-80	Net	7.5	4.07;	4.11;	4.07			4.033	0.735	٠., ••	0.636
4-10-80	Wet	8.0	3.76;	3.75;	3.75			3.753	0.793		0.691
4-10-60	Wet	8.5	3.63;	3.64;	3.64			3.637	0.825	:	0.714
4-10-80	Wet	0.6	3.31;	3.36;	3.35			3,340	868°O		0.777
4-11-80	4-11-80 7.0 Dry + 5.1	5.1 Wet	0.70;	0.70;	0.695; 0.70;	0.70;	0.70	0.699	4.286	,	3.761
4-11-80	4-11-80 6.5 Dry + 8.75	8.75 Wet	0.705;	0.705; 0.7025; 0.700; 0.700; 0.705	0.700;	0.700;	0.705	0.7025	4.270		3.747
!			,							•	

*The times for only 0.5 liters of air were measured for Wet Leg Rotameter Readings of 2.5 through 6.5. Were then multiplied by six to calculate Vm.

TABLE 43. SURFACE FINISH AND HARDNESS OF TEST CYLINDERS

Cylinder number	Surface finish,	Rockwell-C hardness
108	5-12	20
127	4-9	16
147	4-9	21
Specification:	4-9	20-22

ferrite, pearlite, and iron carbide. Cylinder 108 showed carbides not as evenly dispersed as in 127 but more large areas of ferrite. Cylinder 127 showed more carbide than did Cylinder 108.

BOC Evaluation Study

A total of 26 BOC test runs were conducted on clay treated JP-4 to evaluate the general repeatability of the test method. Three different test cylinders, 25 different test balls, and two different percent relative humidity conditions were used in the study. The test cylinders and balls were thoroughly cleaned in alcohol, acetone, and hexane; dried in a vacuum oven in the presence of silica gel; and then stored in a desiccator prior to use. The shaft supporting the test cylinders was thoroughly cleaned and dried between test runs to eliminate the possibility of contamination from one run to the next. The test results are shown in Table 44. A statistical analysis of the test data is shown in Table 45.

Examination of the test data presented in Tables 44 and 45 leads to the following observations and conclusions:

Test Temp., °F Froom Bath 13			12 TO TOOL DA		TABLE AA	中でです	4 F	FROM REPT.	PEPTICATED BO	ROC ETINS		
Run Cylinder Ball Number Number <th>Run Cylinder Ball Toet Temp. °F Hajor Hinor 1 4-14-80 88 105A 10 69-5 73 1.07 0.94 4 4-14-80 88 105A 10 69-5 73 1.07 0.94 5 4-14-80 88 105E 10 66 77 0.94 0.99 6 4-15-80 88 106C 10 66 77 0.94 0.89 7 4-14-80 88 106C 10 66 77 0.61 0.57 6 4-15-80 88 106C 10 66-5 73 0.61 0.57 7 4-14-80 88 106B 20 66-5 72 0.66 0.77 10 4-15-80 88 106B 20 66-5 75 0.66 0.76 11 4-16-80 86 106B 20 66-7 76 0.76 <t< th=""><th></th><th></th><th></th><th></th><th></th><th>-</th><th></th><th></th><th>Koar</th><th>Scar Diago</th><th>1</th></t<></th>	Run Cylinder Ball Toet Temp. °F Hajor Hinor 1 4-14-80 88 105A 10 69-5 73 1.07 0.94 4 4-14-80 88 105A 10 69-5 73 1.07 0.94 5 4-14-80 88 105E 10 66 77 0.94 0.99 6 4-15-80 88 106C 10 66 77 0.94 0.89 7 4-14-80 88 106C 10 66 77 0.61 0.57 6 4-15-80 88 106C 10 66-5 73 0.61 0.57 7 4-14-80 88 106B 20 66-5 72 0.66 0.77 10 4-15-80 88 106B 20 66-5 75 0.66 0.76 11 4-16-80 86 106B 20 66-7 76 0.76 <t< th=""><th></th><th></th><th></th><th></th><th></th><th>-</th><th></th><th></th><th>Koar</th><th>Scar Diago</th><th>1</th></t<>						-			Koar	Scar Diago	1
1 4-14-80 88 105A 10 69-5 73 1.07 0.94 0.83 4 4-14-80 88 105E 10 67 73 0.94 0.83 8 4-15-80 88 106E 10 66 77 0.94 0.83 1 4-15-80 88 106E 10 66 77 0.61 0.51 2 4-14-80 88 105E 20 73.5 0.61 0.51 1 4-15-80 88 105E 20 66.5 72 0.65 0.52 0.65 0.65 0.61 0.52 0.65 0.72 0.65 0.65 0.72 0.65 0.72 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.65 0.75 0.75	1 4-14-80 88 105h 10 69-5 73 1.07 0.94 0.84 4 4-14-80 88 105b 10 67 73 1.07 0.94 0.83 8 4-15-80 88 106c 10 66 77 0.84 0.87 2 4-15-80 88 106c 10 66 76 0.81 0.57 3 4-15-80 88 105c 20 66 76 0.81 0.57 6 4-15-80 88 106c 20 66-5 72 0.81 0.75 10 4-15-80 88 106c 20 66-5 72 0.86 0.75 10 4-15-80 88 106c 20 66-5 72 0.86 0.75 11 4-15-80 88 106c 20 66-5 76 0.86 0.75 11 4-15-80 20 20		Run Sequence	Date	Cylinder Number	Ball Number	SRH	ROOM	Bath		Mirior	Average
4 4-14-90 88 105D 10 67 73 0.94 0.83 5 4-14-80 88 106E 10 67 73 0.94 0.83 6 4-15-80 88 106C 10 66 77 0.81 0.53 2 4-14-80 88 106A 20 68 73 0.81 0.72 0.83 3 4-14-80 88 106A 20 68 73 0.81 0.72 0.85 0.75 0.75 0.86 0.77 0.85 0.77 0.85 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.86 0.77 0.78 0.84 <td>4 4-14-40 88 105D 10 67 73 0.94 0.83 5 4-14-80 88 106E 10 66 77 0.94 0.83 8 4-15-80 88 106D 10 66 77 0.61 0.53 2 4-14-80 88 105C 20 66 73 0.61 0.53 3 4-14-80 88 105C 20 66 73 0.81 0.72 4 15-80 88 106A 20 66 78 0.86 0.75 10 4-15-80 88 106E 20 66 78 0.75 0.66 11 4-18-80 202 107A 10 71.5 78.5 0.76 0.75 14 4-18-80 202 107A 10 69.5 78.5 0.76 0.75 15 4-18-80 202 109C 11 69.5 7</td> <td></td> <td>-</td> <td>4-14-80</td> <td>88</td> <td>105A</td> <td>01</td> <td>69.5</td> <td>73</td> <td>1.07</td> <td>0.94</td> <td>1.01</td>	4 4-14-40 88 105D 10 67 73 0.94 0.83 5 4-14-80 88 106E 10 66 77 0.94 0.83 8 4-15-80 88 106D 10 66 77 0.61 0.53 2 4-14-80 88 105C 20 66 73 0.61 0.53 3 4-14-80 88 105C 20 66 73 0.81 0.72 4 15-80 88 106A 20 66 78 0.86 0.75 10 4-15-80 88 106E 20 66 78 0.75 0.66 11 4-18-80 202 107A 10 71.5 78.5 0.76 0.75 14 4-18-80 202 107A 10 69.5 78.5 0.76 0.75 15 4-18-80 202 109C 11 69.5 7		-	4-14-80	88	105A	01	69.5	73	1.07	0.94	1.01
5 4-14-80 66 17 73 1.02 0.89 8 4-15-80 88 106c 10 66 77 0.84 0.57 9 4-15-80 88 106c 10 66 77 0.81 0.57 1 4-14-80 88 105c 20 66.5 72 0.81 0.75 1 4-15-80 88 106A 20 66.5 72 0.86 0.75 1 4-15-80 88 106A 20 66.5 72 0.86 0.75 1 4-15-80 88 106A 20 66.5 76 0.86 0.76 1 4-15-80 88 106E 20 66 76 0.86 0.75 1 4-18-80 202 107A 10 71.5 78.5 0.86 0.75 1 4-18-80 202 109B 10 69.5 78.5 0.61 <t< td=""><td>5 4-14-80 38 105E 10 67 73 1.02 0.89 8 4-15-80 88 106C 10 66 77 0.84 0.57 9 4-15-80 88 105B 20 73 73 0.81 0.57 1 4-14-80 88 105C 20 68 73 0.81 0.77 1 4-15-80 88 106B 20 68 73 0.86 0.77 1 4-15-80 88 106B 20 66 76 72 0.86 0.77 1 4-15-80 88 106B 20 66 76 0.86 0.77 1 4-15-80 88 106B 20 66 76 0.86 0.77 1 4-18-80 202 107A 10 66-5 76-5 0.86 0.75 1 4-18-80 202 109B 10 69-5</td><td></td><td>1 4</td><td>4-14-80</td><td>39</td><td>105D</td><td>01</td><td>67</td><td>73</td><td>0.94</td><td>0.83</td><td>0.89</td></t<>	5 4-14-80 38 105E 10 67 73 1.02 0.89 8 4-15-80 88 106C 10 66 77 0.84 0.57 9 4-15-80 88 105B 20 73 73 0.81 0.57 1 4-14-80 88 105C 20 68 73 0.81 0.77 1 4-15-80 88 106B 20 68 73 0.86 0.77 1 4-15-80 88 106B 20 66 76 72 0.86 0.77 1 4-15-80 88 106B 20 66 76 0.86 0.77 1 4-15-80 88 106B 20 66 76 0.86 0.77 1 4-18-80 202 107A 10 66-5 76-5 0.86 0.75 1 4-18-80 202 109B 10 69-5		1 4	4-14-80	39	105D	01	67	73	0.94	0.83	0.89
8 4-15-80 88 106C 10 66 77 0.84 0.57 9 4-15-80 88 106D 10 66 76 76 0.61 0.53 3 4-15-80 88 105C 20 66.5 73.5 0.81 0.72 4 4-15-80 88 106B 20 66.5 76 0.86 0.77 10 4-15-80 88 106B 20 66.5 76 0.86 0.77 11 4-15-80 88 106B 20 66.5 76 0.86 0.77 11 4-15-80 88 106B 20 76.5 78.5 0.76 0.77 11 4-18-20 202 107A 10 69.5 78.5 0.65 0.75 15 4-18-20 202 109D 10 69.5 77.5 0.69 0.61 17 4-18-20 202 109D 10 <td>8 4-15-80 88 106C 10 66 77 0.84 0.57 9 4-15-80 88 106D 10 66 76 76 0.61 0.53 2 4-14-80 89 105C 20 66 73 0.72 0.65 3 4-15-80 88 106R 20 66 73 0.72 0.65 10 4-15-80 88 106R 20 66 76 0.86 0.75 1 4-15-80 88 106R 20 66 76 0.86 0.75 1 4-15-80 88 106R 20 66-5 76 0.86 0.75 1 4-15-80 88 106R 20 66-5 76 0.86 0.75 1 4-18-20 202 109A 10 69 78-5 0.71 0.67 1 4-18-20 202 109C 10 69-5<td></td><td>·vn</td><td>4-14-80</td><td>88</td><td>105E</td><td>61</td><td>67</td><td>73</td><td>1.02</td><td>0.89</td><td>96.0</td></td>	8 4-15-80 88 106C 10 66 77 0.84 0.57 9 4-15-80 88 106D 10 66 76 76 0.61 0.53 2 4-14-80 89 105C 20 66 73 0.72 0.65 3 4-15-80 88 106R 20 66 73 0.72 0.65 10 4-15-80 88 106R 20 66 76 0.86 0.75 1 4-15-80 88 106R 20 66 76 0.86 0.75 1 4-15-80 88 106R 20 66-5 76 0.86 0.75 1 4-15-80 88 106R 20 66-5 76 0.86 0.75 1 4-18-20 202 109A 10 69 78-5 0.71 0.67 1 4-18-20 202 109C 10 69-5 <td></td> <td>·vn</td> <td>4-14-80</td> <td>88</td> <td>105E</td> <td>61</td> <td>67</td> <td>73</td> <td>1.02</td> <td>0.89</td> <td>96.0</td>		·vn	4-14-80	88	105E	61	67	73	1.02	0.89	96.0
2 4-15-80 88 106B 10 66 75 73.5 0.61 0.53 3 4-14-80 88 105B 20 68 73 0.81 0.72 4 4-14-80 88 106A 20 66.5 72 0.86 0.77 1 4-15-80 88 106A 20 66.5 72 0.86 0.77 1 4-15-80 88 106A 20 66.5 72 0.86 0.77 1 4-15-80 88 106B 20 66.5 72 0.86 0.77 0.65 1 4-15-80 202 107A 10 71.5 75.5 0.76 0.75 15 4-18-20 202 109B 10 69.5 73.5 0.64 0.51 16 4-18-20 202 109B 10 69.5 73.5 0.64 0.51 17 4-18-80 202 109B <td>2 4-15-80 88 106D 10 66 76 0.61 0.53 2 4-14-80 98 105B 20 70.5 73.5 0.81 0.72 3 4-14-80 88 105C 20 66 73 0.86 0.77 7 4-15-80 88 106B 20 66 76 0.86 0.77 10 4-15-80 88 106B 20 66 76 0.86 0.77 10 4-15-80 88 106B 20 66 78.5 0.76 0.76 11 4-16-80 202 107A 10 71.5 75.5 0.86 0.77 15 4-18-20 202 109B 10 69.5 78.5 0.61 0.51 16 4-18-20 202 109B 10 69.5 78.5 0.61 0.51 17 4-18-20 202 109B 10 69.5<td></td><td>, co</td><td>4-15-80</td><td>88</td><td>106C</td><td>01</td><td>99</td><td>11</td><td>0.84</td><td>0.57</td><td>0.71</td></td>	2 4-15-80 88 106D 10 66 76 0.61 0.53 2 4-14-80 98 105B 20 70.5 73.5 0.81 0.72 3 4-14-80 88 105C 20 66 73 0.86 0.77 7 4-15-80 88 106B 20 66 76 0.86 0.77 10 4-15-80 88 106B 20 66 76 0.86 0.77 10 4-15-80 88 106B 20 66 78.5 0.76 0.76 11 4-16-80 202 107A 10 71.5 75.5 0.86 0.77 15 4-18-20 202 109B 10 69.5 78.5 0.61 0.51 16 4-18-20 202 109B 10 69.5 78.5 0.61 0.51 17 4-18-20 202 109B 10 69.5 <td></td> <td>, co</td> <td>4-15-80</td> <td>88</td> <td>106C</td> <td>01</td> <td>99</td> <td>11</td> <td>0.84</td> <td>0.57</td> <td>0.71</td>		, co	4-15-80	88	106C	01	99	11	0.84	0.57	0.71
2 4-14-80 98 1058 20 70.5 73.5 0.81 0.72 3 4-14-80 88 105C 20 66.5 72 0.86 0.77 7 4-15-80 88 106A 20 66.5 72 0.86 0.77 10 4-15-80 88 106B 20 66.5 76 0.86 0.77 0.65 11 4-15-80 86 106E 20 66.5 76.5 0.86 0.76 0.76 14 4-18-80 202 107A 10 69.5 78.5 0.78 0.67 15 4-18-20 202 109B 10 69.5 78.5 0.64 0.57 16 4-18-20 202 109C 11 69.5 77.5 0.69 0.61 17 4-18-20 202 112A 20 69.5 77.5 0.60 0.61 18 4-16-80 202	2 4-14-80 98 1058 20 70.5 73.5 0.81 0.72 3 4-14-80 88 106A 20 66.5 72 0.86 0.77 6 4-15-80 88 106A 20 66.5 72 0.86 0.77 10 4-15-80 88 106B 20 66.5 72 0.86 0.77 10 4-15-80 88 106B 20 66.5 78.5 0.86 0.77 11 4-16-80 202 107A 10 71.5 75.5 0.87 0.67 14 4-18-20 202 109B 10 69.5 78.5 0.71 0.67 16 4-18-20 202 109B 10 69.5 78.5 0.61 0.51 16 4-18-20 202 112A 10 69.5 78.5 0.62 0.61 18 4-21-80 202 112A 10		6	4-15-80	88	106D	70	99	76	0.61	0.53	0.57
3 4-14-80 88 105c 20 68 73 0.72 0.65 6 4-15-80 88 106A 20 66.5 72 0.86 0.77 7 4-15-80 88 106B 20 66.5 75 0.86 0.77 10 4-15-80 88 106B 20 66 76 0.86 0.77 11 4-15-80 202 107A 10 71.5 75.5 0.86 0.75 14 4-18-80 202 109A 10 69.5 78.5 0.69 0.71 0.62 15 4-18-20 202 109C 10 69.5 78.5 0.64 0.51 17 4-18-20 202 109C 10 69.5 78.5 0.69 0.61 18 4-21-80 202 112A 10 69.5 77.5 0.69 0.61 13 4-16-80 202 107A <	3 4-14-80 88 105C 20 68 73 0.72 0.65 6 4-15-80 88 106A 20 66.5 72 0.86 0.77 1 4-15-80 88 106B 20 66.5 72 0.86 0.77 10 4-15-80 88 106E 20 66 76 0.86 0.77 0.76 11 4-15-80 202 107A 10 71.5 75.5 0.77 0.67 14 4-18-20 202 109A 10 69 78.5 0.71 0.67 15 4-18-20 202 109C 10 69 78.5 0.64 0.57 16 4-18-20 202 109D 10 69.5 78.5 0.64 0.57 17 4-18-20 202 109D 10 69.5 77.5 0.60 0.64 18 4-16-80 202 112A 1		7	4-14-80	88	1,058	20	70.5	73.5	0.81	0.72	0.77
6 4-15-80 88 106A 20 66.5 72 0.86 0.77 7 4-15-80 88 106B 20 66 76 0.86 0.76 10 4-15-80 88 106B 20 66 78.5 0.76 0.76 11 4-16-80 202 107A 10 71.5 75.5 0.75 0.67 14 4-18-20 202 109B 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69.5 78.5 0.61 0.61 16 4-18-20 202 109D 10 69.5 78.5 0.62 0.61 17 4-18-20 202 112A 10 69.5 77.5 0.69 0.61 18 4-21-80 202 112A 20 77.5 0.69 0.61 0.61 19 4-16-80 202 107B 20	6 4-15-80 88 106A 20 66.5 72 0.86 0.77 7 4-15-80 88 106B 20 66 76 76 0.86 0.77 10 4-15-80 88 106B 20 66 76 76 0.86 0.77 11 4-18-80 202 107A 10 71.5 75.5 0.71 0.67 14 4-18-20 202 109A 10 69 78.5 0.71 0.62 15 4-18-20 202 109C 10 69 78.5 0.64 0.51 16 4-18-20 202 109D 10 69.5 78 0.63 0.54 17 4-18-20 202 112A 10 69.5 77.5 0.69 0.61 18 4-16-80 202 107A 20 77.5 0.69 0.61 0.61 19 4-16-80 202 107		. m	4-14-80	8	105C	70	89	73	0.72	0.65	69.0
7 4-15-80 88 1068 20 66 76 76 0.96 6.76 10 4-15-80 88 106E 20 66 78.5 0.76 0.76 11 4-15-80 202 107A 10 71.5 75.5 0.85 0.67 14 4-18-80 202 109A 10 69.5 78.5 0.71 0.67 15 4-18-20 202 109C 1. 69 78.5 0.69 0.71 0.62 16 4-18-20 202 109C 1. 69 78.5 0.69 0.61 0.51 17 4-18-20 202 109C 1. 69.5 78 0.69 0.61 0.51 18 4-21-80 202 112A 10 69.5 77 0.69 0.61 13 4-16-80 202 107A 20 77 7 0.69 0.61 13 4-21	7 4-15-80 88 1068 20 66 76 0.96 6.76 10 4-15-80 88 106E 20 66 78.5 0.76 0.76 11 4-15-80 202 107A 10 71.5 75.5 0.85 0.71 14 4-18-80 202 109A 10 69.5 78.5 0.85 0.71 15 4-18-20 202 109B 10 69.5 78.5 0.60 0.71 0.62 16 4-18-20 202 109D 10 69.5 78.5 0.60 0.71 0.63 16 4-18-20 202 109D 10 69.5 78.5 0.64 0.51 18 4-21-80 202 107A 20 77.5 0.69 0.61 0.61 13 4-16-80 202 107B 20 107B 77.5 78 0.69 0.78 0.61 13		9	4-15-80	88	106A	20	66.5	72	0.86	0.77	0.82
10 4-15-80 88 106E 20 66 78.5 0.76 0.67 11 4-16-80 202 107A 10 71.5 75.5 0.85 0.75 14 4-18-80 202 109B 10 69.5 78.5 0.69 0.71 0.62 15 4-18-20 202 109D 10 69.5 78.5 0.69 0.61 0.51 17 4-18-20 202 109D 10 69.5 78.5 0.69 0.51 18 4-18-20 202 107B 10 69.5 79.5 0.69 0.51 18 4-18-80 202 112A 10 69.5 77.5 0.60 0.51 13 4-16-80 202 107B 20 77.5 0.60 0.61 13 4-16-80 202 112B 20 76.5 78 0.62 0.62 20 4-21-80 202 112B<	10 4-15-80 88 106E 20 66 78.5 0.76 0.67 11 4-16-80 202 107A 10 71.5 75.5 0.85 0.75 14 4-18-80 202 109A 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69 78.5 0.69 0.61 16 4-18-20 202 109D 10 69 78.5 0.69 0.61 17 4-18-20 202 109D 10 69.5 78 0.69 0.61 18 4-21-80 202 112A 10 69.5 77 0.69 0.61 13 4-16-80 202 107A 20 74 77 0.69 0.61 13 4-16-80 202 107B 20 76.5 78 0.62 0.51 20 4-21-80 202 112B 20 <		7	4-15-80	33	1068	20	99	76	0.86	6.76	0.81
11 4-16-80 202 107A 10 71.5 75.5 0.85 0.75 14 4-18-80 202 109A 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69.5 78.5 0.69 0.61 16 4-18-20 202 109D 10 69.5 78.5 0.69 0.61 17 4-18-20 202 109D 10 69.5 78 0.63 0.51 18 4-21-80 202 112A 10 69.5 77.5 0.60 0.51 13 4-16-80 202 112A 20 77.5 0.60 0.51 19 4-16-80 202 117B 20 76.5 78.5 0.62 0.62 20 4-21-80 202 112B 20 70.5 78 0.78 0.65 21 4-22-80 202 113A 20 70.5 </td <td>11 4-16-80 202 107A 10 71.5 75.5 0.085 0.75 14 4-18-80 202 109A 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69 78.5 0.69 0.61 0.61 0.62 16 4-18-20 202 109D 10 69.5 78 0.69 0.61 0.61 0.62 0.61 0.62 0.61 0.62 0.61 0.62 0.61 0.62 0.62 0.61 0.62 0.62 0.61 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.63 0.62 0.62 0.62 0.62 0.62 0.63 0.64 0.54 0.64 0.54 0.62 0.62 0.61 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 <</td> <td></td> <td>01</td> <td>4-15-80</td> <td>88</td> <td>106E</td> <td>20</td> <td>99</td> <td>78.5</td> <td>0.76</td> <td>0.67</td> <td>0.72</td>	11 4-16-80 202 107A 10 71.5 75.5 0.085 0.75 14 4-18-80 202 109A 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69 78.5 0.69 0.61 0.61 0.62 16 4-18-20 202 109D 10 69.5 78 0.69 0.61 0.61 0.62 0.61 0.62 0.61 0.62 0.61 0.62 0.61 0.62 0.62 0.61 0.62 0.62 0.61 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.63 0.62 0.62 0.62 0.62 0.62 0.63 0.64 0.54 0.64 0.54 0.62 0.62 0.61 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 0.62 <		01	4-15-80	88	106E	20	99	78.5	0.76	0.67	0.72
14 4-18-80 202 109A 10 69.5 78.5 0.71 0.62 15 4-18-20 262 109B 10 69 78.5 0.69 0.61 16 4-18-20 202 109C 1J 69 78.5 0.69 0.61 17 4-18-20 202 109D 10 69.5 78 0.69 0.51 18 4-21-80 202 112A 10 69.5 7/7 0.60 0.51 13 4-16-80 202 107A 20 74 77 0.60 0.51 19 4-21-80 202 107B 20 76.5 78.5 0.60 0.51 20 4-21-80 202 112B 20 70.5 78 0.78 0.68 21 4-21-80 202 113B 20 70.5 76 0.60 0.51 22 4-22-80 202 113B 20	14 4-18-80 202 1094 10 69.5 78.5 0.71 0.62 15 4-18-20 202 109B 10 69 78.5 0.69 0.61 16 4-18-20 202 109C 10 69 78.5 0.69 0.61 17 4-18-20 202 109D 10 69.5 79 0.69 0.61 18 4-21-80 202 112A 10 69.5 77.5 0.60 0.51 12 4-16-80 202 107A 20 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 13 4-16-80 202 112B 20 76.5 78 0.69 0.61 20 4-21-80 202 112B 20 76.5 78 0.76 21 4-22-80 202 113A 20 70.5 76 <t< td=""><td></td><td>=</td><td>4-16-80</td><td>202</td><td>1078</td><td>10</td><td>71.5</td><td>75.5</td><td>0.85</td><td>0.75</td><td>98.0</td></t<>		=	4-16-80	202	1078	10	71.5	75.5	0.85	0.75	98.0
15 4-18-20 262 109B 10 69 78.5 0.69 0.61 16 4-18-20 202 109C 1J 69 78.5 0.64 0.57 17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-16-80 202 112A 10 69.5 77.5 0.60 0.51 13 4-16-80 202 107A 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 76.5 78.5 0.69 0.51 20 4-21-80 202 112B 20 70.5 78 0.78 0.69 21 4-21-80 202 112D 20 70.5 78 0.78 0.76 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 202 113B 20	15 4-18-20 262 109B 10 69 78.5 0.69 0.61 16 4-18-20 202 109C 1J 69 78.5 0.64 0.57 17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-21-80 202 11ZA 10 69.5 77.5 0.60 0.51 12 4-16-80 202 11ZA 20 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 76 6.69 0.69 20 4-21-80 202 112B 20 70.5 78 0.69 0.69 21 4-21-80 202 112D 20 70.5 76 0.60 0.76 22 4-22-80 202 113A 20 70.5 75.5 <t< td=""><td></td><td>11</td><td>4-18-80</td><td>202</td><td>109A</td><td>01</td><td>69.5</td><td>78.5</td><td>0.71</td><td>0.62</td><td>0.67</td></t<>		11	4-18-80	202	109A	01	69.5	78.5	0.71	0.62	0.67
16 4-18-20 202 109C 1J 69 78.5 0.64 0.57 17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-21-80 202 112A 10 69.5 7;.5 0.60 0.51 13 4-16-80 202 107A 20 74 77 0.92 0.81 19 4-21-80 202 117B 20 70.5 78.5 0.69 0.55 20 4-21-80 202 112D 20 70.5 78 0.78 0.68 21 4-21-80 202 112D 20 70.5 78 0.78 0.66 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 202 113B 20 75.5 0.75 0.75	16 4-18-20 202 109C 1J 69 78.5 0.64 0.57 17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-16-80 202 112A 20 77 0.92 0.61 13 4-16-80 202 107B 20 74 77 0.69 0.61 13 4-16-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 76 78 0.69 0.66 20 4-21-80 202 112D 20 70.5 78 0.76 0.76 21 4-22-80 202 113A 20 70.5 76 0.66 0.71 22 4-22-80 184 113C 20 71 77 0.73 0.66		15	4-18-20	202	1098	10	69	78.5	0.69	0.61	0.65
17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-21-80 202 112A 10 69.5 77.5 0.60 0.51 12 4-16-80 202 107A 20 74 77 0.92 0.81 13 4-21-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 70.5 78.5 0.69 0.65 20 4-21-80 202 112D 20 70.5 78 0.78 0.68 21 4-21-80 202 113A 20 70.5 76 0.60 0.51 22 4-22-80 202 113B 20 75.5 0.55 0.51 23 4-22-80 202 113B 20 75.5 0.73 0.65	17 4-18-20 202 109D 10 69.5 78 0.63 0.54 18 4-21-80 202 112A 10 69.5 77.5 0.92 0.81 12 4-16-80 202 107A 20 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.92 0.81 19 4-21-80 202 112B 20 70.5 78.5 0.62 0.55 20 4-21-80 202 112D 20 70.5 78 0.78 0.76 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 104 113C 20 70.5 75.5 0.73 0.66 24 4-22-80 184 113C 20 71 77 0.73 0.66		16	4-18-20	202	109C	3	69	78.5	0.64	0.57	0.61
12 4-16-80 202 107A 20 72 77 0.92 0.81 12 4-16-80 202 107B 20 74 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 70.5 78.5 0.62 0.55 20 4-21-80 202 112D 20 70.5 78 0.78 0.68 21 4-21-80 202 113A 20 70.5 78 0.86 0.76 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 202 113B 20 75.5 C.73 0.65	12 4-16-80 202 107A 20 72 77 0.92 0.81 12 4-16-80 202 107B 20 74 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 13 4-16-80 202 112B 20 76 77 0.69 0.61 20 4-21-80 202 112C 20 70.5 78 0.69 0.61 21 4-21-80 202 113D 20 70.5 78 0.78 0.68 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 202 113B 20 70.5 75.5 0.73 0.65 24 4-22-80 184 113C 20 71 77 0.73 0.66	1	17	4-18:20	202	109D	01	69.5	78	0.63	0.54	0.59
12 4-16-80 202 107A 20 72 17 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 70.5 78 0.69 0.61 20 4-21-80 202 112C 20 70.5 78 0.78 0.68 21 4-21-80 202 113A 20 70.5 76 0.60 0.71 22 4-22-80 202 113B 20 70.5 75.5 0.73 0.65	12 4-16-80 202 207A 20 72 77 0.92 0.81 13 4-16-80 202 107B 20 74 77 0.69 0.61 19 4-21-80 202 112B 20 70.5 78 0.69 0.61 20 4-21-80 202 112C 20 70.5 78 0.78 0.68 21 4-21-80 202 113A 20 70.5 76 0.60 0.71 22 4-22-80 202 113A 20 70.5 76 0.60 0.51 23 4-22-80 184 113C 20 71 77 0.73 0.66	34	18	4-21-80	202	112A	01	69.5	75.5	0.60	0.51	o. 56
4-16-80 202 107B 20 74 77 0.69 0.61 4-21-80 202 112B 20 70.5 78.5 0.62 0.55 4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 113D 20 70.5 78 0.86 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 75.5 C.73 0.65	4-16-80 202 107B 20 74 77 0.69 0.61 4-21-80 202 112B 20 70.5 78.5 0.62 0.55 4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 112D 20 70.5 78 0.76 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 184 113C 20 71 77 0.73 0.66	ŀ	12	4-16-80	202	107A	20	72	ŗ	0.92	0.81	0.87
4-21-80 202 112B 20 70.5 78.5 0.62 0.55 4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 112D 20 70.5 78 0.86 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 70.5 75.5 C.73 0.65	4-21-80 202 112B 20 70.5 78.5 0.62 0.55 4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 112D 20 70.5 78 0.86 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 184 113C 20 71 77 0.73 0.66		13	4-16-80	202	1078	20	. 7.4	77	0.69	0.61	0.65
4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 112D 20 70.5 78 0.96 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 75.5 C.73 0.65	4-21-80 202 112C 20 70.5 78 0.78 0.68 4-21-80 202 112D 20 70.5 78 0.86 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 73.5 75.5 0.73 0.65 4-22-80 184 113C 20 71 77 0.73 0.66		13	4-21-80	202	1128	70	70.5	78.5	0.62	0.55	0.59
4-21-80 202 112D 20 70.5 78 0.96 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 70.5 75.5 C.73 0.65	4-21-80 202 112D 20 70.5 78 0.86 0.76 4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 73.5 75.5 0.73 0.65 4-22-80 184 113C 20 71 77 0.73 0.66		50	4-21-90	202	112C	20	70.5	78	0.78	0.68	0.73
4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 70.5 75.5 C.73 0.65	4-22-80 202 113A 20 70.5 76 0.60 0.51 4-22-80 202 113B 20 70.5 75.5 0.73 0.65 4-22-80 184 113C 20 71 77 0.73 0.66		21	4-21-80	202	1120	70	70.5	78	98.0	0.76	0.81
4-22-80 202 113B 20 70.5 75.5 C.73 0.65	4-22-80 202 113B 20 70.5 75.5 0.73 0.65 4-22-80 184 113C 20 71 77 0.73 0.66		22	4-22-80	202	113A	50	70.5	26	09.0	0.51	0.56
	4-22-80 184 113C 20 71 77 0.56		23	4-22-80	202	1138	70	70.5	75.5	C.73	0.65	69.0
	בלים כפיס אבי ער ערור זפו ספיכרי					1111	2 6		7	6	2,2	7.7

TABLE 45. PRECISION CALCULATIONS FOR BOC TEST DATA

Test	parameters	compare	ed		Average wear	σOf
Cylinder	number	% RH	Runs	Days	diameter, mm	average
88		10	5	2	0.83	0.18
88		20	5	2	0.76	0.06
202		10	6	3	0.65	0.09
202		20	7	4	0.70	0.11
184		20	3	1	0.69	0.09
88		10, 20	10	2	0.80	0.13
202		10, 20	13	4	0.68	0.10
88, 20	02	10	11	5	0.73	0.16
88, 202,	184	20	15	5	0.72	0.09
A11	•	10, 20	26	6	0.72	0.12

- (a) The percent relative standard deviation for the average of all 26 runs was ±17%. This precision was approximately the same whether only one or multiple test cylinders were used.
- (b) The overall precision for the runs at 20% RH (0.09 σ) was significantly better than the precision at 10% RH (0.16 σ).
- (c) It was suspected that a major part of the precision problem may have been due to nonhomogeneity of the test balls. This may explain the lack of a consistent difference in ball wear between the major and minor axis.
- (d) More test data should be generated to evaluate the effects of relative humidity and ball variation on test precision.
- 16. <u>DETERMINATION OF PRECISION FOR THE SIMULATED DISTILLATION ANALYSIS, ASTM D 2887</u>

Background

Simulated distillation analyses by gas chromatography were conducted on 19 blind repeat fuel samples which had been analyzed earlier at times that varied from 3 to 10 months. The repeat

samples were not the same samples analyzed earlier but were fresh samples taken from the same drums of fuel. Since significant differences occurred in some of the replicated test results, the data were evaluated to determine whether specific trends and causes could be identified.

Evaluation of Test Data

The test data for nineteen pairs of duplicate samples are compared in Table 46. Table 47 shows differences in selected percent recovery values for the second set of data as compared to the first. This is essentially a measure of reproducibility since different samples were analyzed at different times against different calibration curves. The chromatograms and computer data processing charts were evaluated to insure there were no obvious errors in any of the analyses and then the data in Tables 46 and 47 were studied to enable some conclusions to be drawn about the test result variations. The ASTM method allows the following reproducibility values: 8.3°C for initial boiling point, 3.3°C for 5% recovered, 3.9°C for 10% recovered, 4.5°C for 50% recovered, 5.6°C for 95% recovered, and 13.4°C for final boiling point.

Conclusions

- (a) With the exception of initial boiling point, most of the test data (particularly final boiling points) fell within the acceptable ASTM guidelines for reproducibility.
- (b) Because of the extended length of time between repeat analyses, the variations in our test results would be expected to be larger than those encountered by a single laboratory conducting duplicate analyses of the same fuel.

DUPLICATE SIMULATED DISTILLATIONS OVER VARIOUS TIME SPANS TABLE 46.

The state of the s	•							1	mich	INGIC	200	rcents	e mere	Recovered	100							
COLL NUMBER		٦,	_	7.		<u> </u>	`r	-	5		,	2	-		L	-	•	-	F	ľ	ŀ	Γ
Mos. Retween Runs: 13/4	18: 1	3/4	1	4/4	1,	(4	1,	•	13/	•	13		_	-	Š	-	,_	-	T.		1	
cample Numbers:	: S3A	80-1	34A	80-2	YBY	30-3	¥68	80-4	\$56A	8-5	S57A	80-6	900	80-7	02	8-08	900	6-08	94	97-8	AXO.	1
Percents			L														1	7	1	1		
0.5 (IMP)	139	140	139	145	7	53	77	93	101	118	36		128 1				8	8	31	*	32	\$
. 4	151	151	121	157	28	37	85	111	127	136	120	133		150	145	151	76	8	7	2	\$	57
2	184	182	184	188	5	99	156	162	170	170	167							7	16	2	2	2
21	199	197	199	204	81	98	178	178	164	163	102 1			_		_		_		901	10	*
70	219	218	219	526	99	103	195	195	861	197										501	123	•
30	236	232	236	241	120	119	50 6	205	209	502						***		_		_	597	139
40	251	248	157	255	140	134	216	216	218	917	21.7 2			_		_			•		191	153
20	563	250	263	569	191	151	223	222	225	225			231 2					176	166	\$3	991	191
09	276	273	275	283	182	173	232	222	236	234			·	_		_			-		0.1	351
7.	290	286	289	599	201	193	240	240	242	244	•			_				_		_	2	2
90	305	304	505	315	219	215	252	250	254	255								_			20	S
8	325	329	328	340	238	236	263	263	766	270	265 2	_		_	•	_		_			232	232
95	344	347	343	360	254	253	27.1	270	274	285										247	,	252
8	175	171	373	386	27.1	271	286	273	295	26	292 3			-				-		22	200	200
99.5 (PBP)	380	375	379	391	1 279	276	290	280	302	307			.,	_			301	_			6	282
										-				•		-		•		-		-

			CR014		150	164	190	203	225	234	250	262	276	162	308	328	343	362	364
		 -	241		124	152	186	204	227	232	250	262	278	295	311	332	348	381	388
			CRF13		147	162	193	211	234	253	768	282	295	308	324	347	998	386	399
	٤	200	130		139	150	194	211	236	254	569	282	296	308	322	342	359	399	407
Recovered			CR08		38	8	62	83	142	179	195	211	225	232	247	263	278	302	306
		•	ည္ထ		4	53	67	8	166	189	206	218	230	234	250	264	281	322	330
ts Were			BRE9		8	9	\$	68	101	120	143	167	1.89	210	230	253	273	302	307
Percents	14.	4	86		52	36	17	68	105	176	150	174	198	218	232	253	267	294	301
C at Which Indicated			BRF14		140	154	162	196	218	230	246	258	273	167	312	338	360	38	4 0 4
	1	7	148		122	153	178	196	216	231	242	255	271	287	306	337	351	393	404
			BRF13		138	151	181	199	221	241	256	271	287	304	319	344	363	392	397
	-		138		121	141	111	196	222	240	255	271	287	302	318	340	362	400	409
	3	7	8-13		146	159	186	199	218	234	247	259	272	287	364	323	349	375	379
	-	7	DF2		132	147	180	197	216	231	245	256	271	284	300	318	333	343	373
	12	7	8-13		34	42	9/	92	117	133	154	171	192	216	244	288	313	344	352
			4GX		28	36	78	86	121	143	164	178	204	230	264	301	318	342	346
	Pair Number:	Mos.Between Runs:	Sample Numbers:	Percents	0.5 (IBP)	-	ĽΩ	70	20	30	40	20	09	20	080	20	95	93	99.5 (FBP)

DIFFERENCES IN RESULTS BETWEEN DUPLICATED SIMULATED DISTILLATIONS TABLE 47.

Matte Devistion Limits ^C		£8.3	£.54 8.54	\$5.6 \$13.4
Merage Devi- ation		#11.3 #8.4	### ### ###	46.6 48.1
Average Net		+11.1	10°0	# C
*C Change in Second Result Compared to the First Result	13/6 13/6 13/6 13/6 13/6 13/6 7 54 7 54 7 7 7 4 4 4 2 2 2 2	+1 +6 +8 +16 +14 +18 +12 +14 +6 +7 +8 +6 +14 +17 +18 +5 -2 +17 +26 0 +6 +9 +19 +9 +13 +6 +6 +2 +13 +17 +6 +12 +10 +1 +4 -3 +12 +12	-2 +4 +4 +6 0 +2 -2 +1 -3 -2 +1 -2 +6 +4 +4 -2 -5 -1 +2 -5 -1 -2 +5 +5 +1 0 0 -1 +2 -4 +1 -4 0 -1 -6 +2 +3 0 0 -8 0 -1 -3 +6 -10 -1 0 +4 -5 +4 -1 -5 -5 -6 +3 0 +3 -7 -7 0 0	+3 +17 -1 -1 +11 +12 +1 +13 xx +1 xx -5 +15 +1 +9 +6 -3 +7 -5 -5 +12 -1 -10 +5 +6 -5 -1 -1 -16 -4 +6 +6 -12 0 +6 -24 -8 -24
	Pair Mumber: Time Span, Mos:	6 Recovered 0.5 (IBP) 1.0	5.0 10.0 50.08	95 99.5 (PBP)

B Useful for determining randomness of deviations.

Without regard to sign.

- (c) The fact that the initial boiling points were almost always higher for the second analysis atrongly suggests that small amounts of the low boiling compounds may have been lost from the fuels during the time period between analyses.
- (d) An examination of the chromatograms for repeat analyses did not reveal gross differences. Most of the deviations in test results with the exception of initial boiling point can be attributed to normal instrumental and experimental variations. However, exceptionally poor reproducibility was obtained for sample pairs 17 and 19. For these pairs, final boiling points differed by 24 cegrees centigrade. Examination of chromatograms and raw data for pair 19 shows that the samples exhibited substantial tailing after the final chromatographic peak. In such cases, accurate determination of final boiling point is more subject to error because of the extremely flat count-rate plot (counts accrued versus retention time).

Recommendations

It was recommended that better controlled experiments be conducted if additional information is required regarding the precision of the simulated distillation analysis. Replicate analyses of the same sample should be conducted at one time to determine method repeatability. Then, several days later the same sample should be reanalyzed against a new set of standards to determine method reproducibility.

Because of the indicated loss of volatiles from the test fuels during storage, as indicated by initial boiling point data, it was recommended that improved methods for long-term storage of fuels should be investigated.

17. CHARACTERIZATION OF BLENDED ERBS FUELS FROM NASA

A large number of physical and chemical properties were determined for four samples of blended ERBS fuels being used by NASA-Lewis Research Center, in tests to evaluate broad specification fuels.

Vapor Pressure, Surface Tension, and Kinematic Viscosity

Vapor pressure, surface tension, and kinematic viscosity were determined as a function of temperature, and these properties are presented in Table 48. Vapor pressure was determined by use of the micro-vapor pressure apparatus and procedure described in (ref. 9). Surface tension was determined by the capillary rise method. Kinematic viscosity was determined by ASTM Method D 445, and the viscosity/temperature plots are presented in Figures 57-60.

Hydrocarbon-Type Analyses by Three Methods

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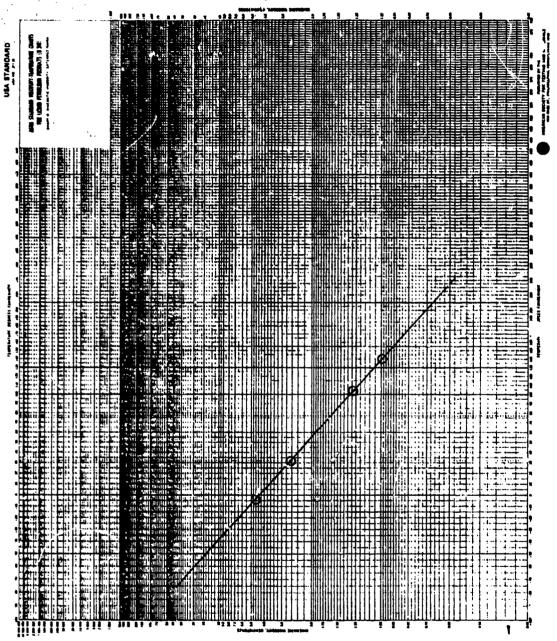
Hydrocarbon type analyses were conducted by three mass spectrometric methods. The first consisted of a separation into paraffinic and aromatic fractions using the procedure described in ASTM D 2549, followed by the mass spectrometric analysis described in ASTM D 2425. The second method used a modification of ASTM D 2789. The third method, Monsanto Method 21-PQ-38-63, was developed for hydrocarbon feed stocks.

The results of all three analyses are presented in Table 49.

TABLE 48. VAPOR PRESSURE, SURFACE TENSION, AND KINEMATIC VISCOSITY

	Temp.,		Test	fuels	
Property	°F	35	3B	3B-11.8	3B-12.3
Vapor Pressure,	32	9.0	8.0	8.0	4.0
num mercury	70	18.0	12.5	14.5	8.0
-	10C	28.5	16.5	22.5	13.5
Surface Tension,	-20 ^a	34.86	32.60	33.02	32.95
dynes/cm	32	32.10	29.79	30.47	30.25
•	70	30.05	27.71	28.59	28.27
	100	28.47	26.07	27.13	26.71
Kinematic Viscosity,	-4	4.626	8.187	6.037	6.920
centistokes	32	2.649	4.186	3.305	3.668
	104	1.286	1.789	1.509	1.633
	140	0.9955	1.331	1.152	1.232

aValues at this temperature were obtained by extrapolation of data from the higher temperatures.



Viscosity/temperature plot for ERBS fuel 3S. Figure 57.

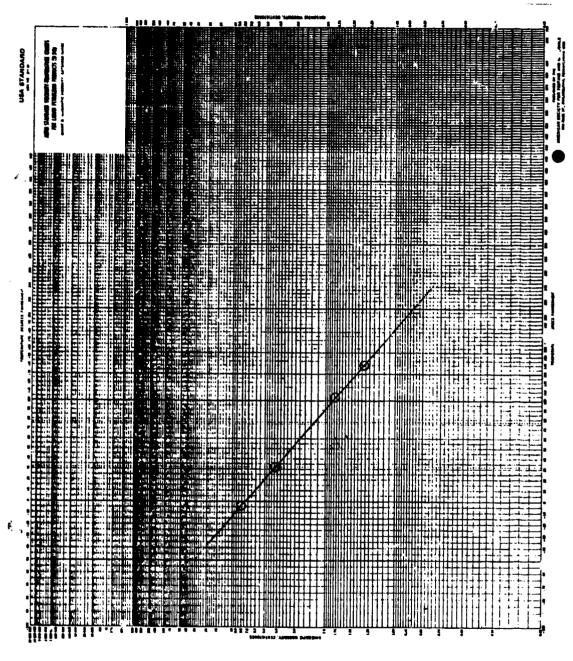
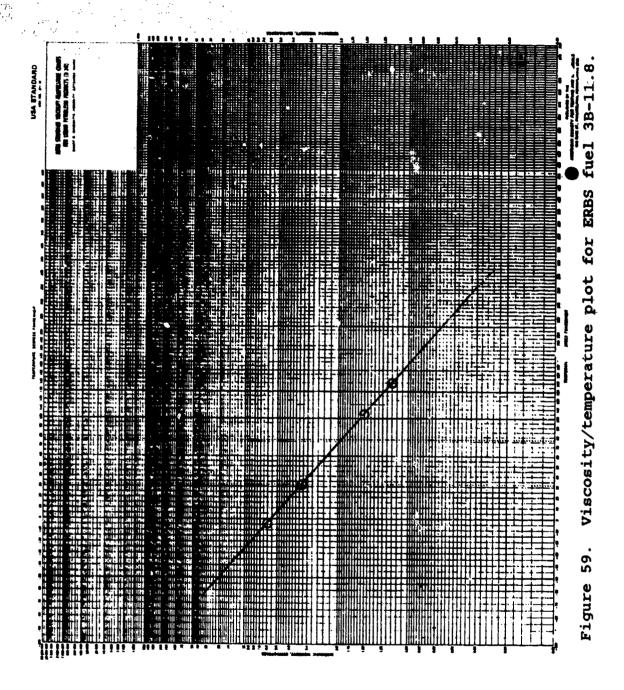
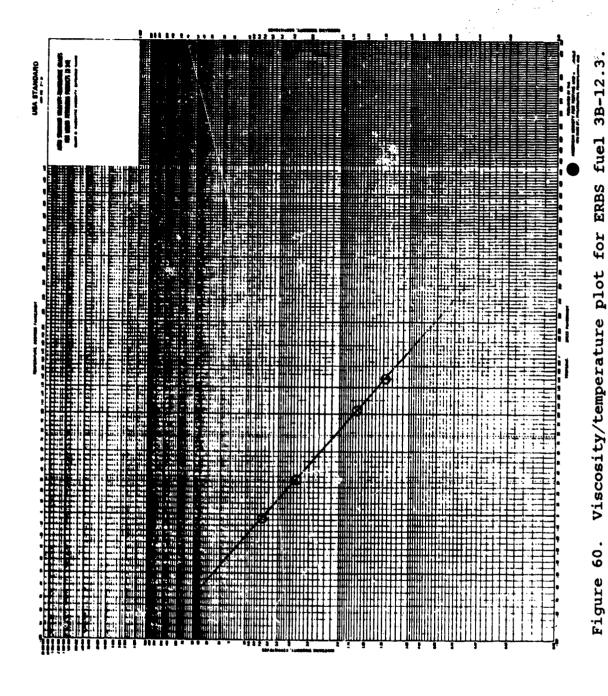


Figure 58. Viscosity/temperature plot for ERBS fuel 3B.





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TABLE 49. HYDROCARBON TYPE ANALYSIS OF THE ERBS FUELS BY THREE METHODS, IN WEIGHT PERCENTS

MOHE	35.1	24.1 20.8 7.0	13.0	
38-12.3 D2789	37.4 22.9	25.6 17.9 8.8	10.3	
D2425	37.7	13.0	12.4 2.1 1.3	60.2 39.8
NOM	30.0	19.8 29.5 6.3	14.4	
3B-11.8 D2789	32. 19.5	21.7 26.3 7.6	11.5	
02425	0.00	21.12		6.3 6.5 8.6
SNOW	42.1	29.3 9.7 7.5	11.4	
3B D2789	23.4 27.5 2.8	80.4 6.04	6	
02425	38.7 14.6 7.2	2 6 6 7 1 1 6 6 6 7 1	12.2 3.0 1.3 1.3	62.2 37.8
MONS	13.9	55.1	19.2	
35 D2789	16.6	5.4. 4.5.6 2.0	16.3	
02425	17.0 3.7 2.9	0 L N N e n L e 1	22 22 23 24 24 24 24 24 24 24 24 24 24 24 24 24	29.8 70.2
Fuel Number: Analytical Method:	Compound Paraffins Monocycloparaffins Dicycloparaffins	Tricycloparaffins Total-Cycloparaffins Alkyl benzenes Indans/tetralins Indenes	Naphthalene Naphthalenes Acenaphthenes Acenaphthylenes Tricyclic aromatics	ASTM D2549 Fractionation wt & aliphatics: wt & aromatics:

^aFor analytical methods, D 2425 designation indicates that fuel was first fractionated according to ASTM D 2549 and then analyzed by ASTM D 2426; D 2789 designation indicates that the fuel was analyzed by a modification of ASTM D 2789 and MONS designation indicates that Monsanto Method 21-PQ-38-63 was used.

brotal of mono-, di-, and tri- cycloparaffins listed above.

CA dash indicates that the compound was included in the analysis but none was detected. A blank space in the table indicates that the compound was not included in the analysis.

Although the results for ASTM Method D 2789 were determined in volume percent, the values in Table 49 are presented in weight percents for the purpose of comparison with the other two methods. The determined volume percents and density values used to calculate weight percents are provided in Table 52 (see the next topic).

For the ASTM D 2425 analysis of Sample 3S, 6.2% of the 35.5% alkylbenzenes appeared in the aliphatic fraction. This fact indicates that there was some breakthrough of aromatics into the aliphatic fraction during the elution chromatographic separation described in ASTM D 2549. Only 0.3% alkylbenzenes appeared in the aliphatic fractions of the other three samples.

Determination of Fuel Aromaticity by NMR and Mass Spectrometry

Scope of Data Provided

Carbon-13 NMR analyses were conducted to determine carbon aromaticity, which is a ratio of aromatic carbon in the fuel to the total carbon. Proton NMR analyses were conducted to determine hydrogen aromaticity, which is a ratio of aromatic hydrogen in the fuel to the total hydrogen. Using these aromaticity values and the ratio of total hydrogen to total carbon (obtained from elemental hydrogen analyses by a wide-line NMR method conducted at AFWAL/POSF), the ratio of aromatic hydrogen to aromatic carbon was then calculated for the four fuels. The total areas under peaks in specified spectral regions of the proton NMR curves are also provided for use in the AFWAL/POSF computer program, if desired.

Carbon aromaticity was then calculated from the ASTM D 2789 hydrocarbon type analysis data in order to compare with the NMR results. An average compound structure was formulated for each fuel constituent using an average carbon number determined from simulated distillation analyses. The percent of aromatic and aliphatic carbon in each fuel constituent was calculated from

the weight percentages and average compound structures. The carbon aromaticity of the entire fuel was then calculated from the ratio of total aromatic carbon to total carbon in the fuel.

Aromaticity by NMR Analyses

The NMR analyses were conducted on a Varian CFT-20 Fourier transform spectrometer containing a Varian 602L computer for data acquisition, data reduction, and system control. The spectrometer was operated at 20 megahertz for the ¹³C analyses and 79.54 megahertz for the proton NMR analyses. The following instrument conditions were utilized for the two analyses:

Type NMR Analysis:	Carbon-13 Proton (1H)
Sample probe Sweep width Number of transients Acquisition time Pulse width 90° Pulse delay Homo-spoil time Data points Decoupler mode	8 mm 5 mm 4,000 Hz 1,000 Hz 1,000 50 1.023 s 4.095 s 17 \(\mu/s\) 5 s 8 s Not on 4 m/s 8,192 3
Chemical shift regions Aromatics for integral data Aliphatics	~150-110 ppm 8.3-6.5 ppm ~70-4 ppm 4.0-0.2 ppm

aHomo-spoil was on during pulse delay.

The analytical samples were prepared for carbon-13 analysis by mixing the following components:

Jet fuel sample - 1 ml

NMR lock solvent - 0.5 ml deuterated chloroform

("100%" CDCl₃)

The gated proton decoupler was on during acquisition and off during delay.

Chemical shift reference - 50 µl hexamethyl disiloxane Relaxation agent - ~25 mg 2,4-pentanedione chromium III derivative

For the proton NMR analysis, a 20 μ l quantity of the above mixture was added to 0.5 ml additional CDCl₃.

The carbon aromaticity values were obtained by integrating the peak areas in the aromatic region of the ¹³C spectra relative to the total peak area in the spectra. The hydrogen aromaticities were obtained in the same manner from peak areas in the proton NMR spectra. The ratio of aromatic hydrogen to aromatic carbon was obtained from the following equation:

$$\frac{H_{ar}}{C_{ar}} = \frac{C_T}{C_{ar}} \times \frac{H_{ar}}{H_T} \times \frac{H_T}{C_T}$$
I II III

where I =the inverse of carbon aromaticity determined by ¹³C NMR

II = the hydrogen aromaticity determined by proton NMR

III = total hydrogen/carbon ratio determined from percent hydrogen data provided by AFWAL/POSF

The aromaticity values for the four fuels and their aromatic and total hydrogen/carbon ratios are presented in Table 50. The integrated areas for the specified spectral regions of the proton NMR spectra are listed in Table 51.

Carbon Aromaticity from Mass Spectrometric Analysis Data

Carbon aromaticity was calculated from the ASTM D 2789 hydrocarbontype analysis data presented in Table 49. Volume percents obtained by this method were re-computed to weight percents using average density values, and an average compound structure was

TABLE 50. AROMATICITY VALUES AND HYDROGEN/CARBON RATIOS

Sample designation	Carbon aromaticity, ^a C _{ar} /C _T	Hydrogen aromaticity, ^a H _{ar} /H _T	Weight percent ^b H _T	Mole ratio ^C H _T /C _T	Mole ratio H _{ar} /C _{ar}
ERBS-35	0.542±0.009	0.244±0.003	10.34	1.37	0.617
TRBS-3B	0.194±0.006	0.072±0.007	12.95	1.77	0.657
3B-11.8	0.355±0.C15	0.135±0.008	11.85	1.60	0.608
3B-12.3	0.273±0.010	0.110±0.003	12.38	1.68	0.677

^aThe reported values represent the average of three integrations per sample.

TABLE 51. INTEGRATED AREAS OF SPECIFIED PROTON NMR SPECTRAL REGIONS

	HMONO 6.6-	HDI 7.3-	HTRI 7.8-	HALP-1 2.3-	HALP-2 1.9-	H BETA 1.9-	H GANA 1.0-	H BETH 1.90-
Samples	7.3, ppm	7.8, ppm	8.3, ppm	4.0, ppm	2.3, ppm	1.0, ppm	0.5, ppm	1.65, ppm
35	16	3	NDp	8	18	22	10	3
3 B	5	2	ND	6	4	45	30	2
3B-11.8	9	3	ND	5	10	37	22	3
3B-12.3	8	2	ND	3	7	40	27	2

The listed integral areas are accurate to ±0.5. Fractional values are rounded to the nearest whole number by the data system.

formulated for each fuel constituent using the average carbon number from a GC simulated distillation analysis. The percents of aromatic and aliphatic carbon in each fuel constituent were calculated from the weight percentages and compound structures. The carbon aromaticity of the entire fuel was then calculated from the ratio of total aromatic carbon to total carbon in the fuel. These results and all data used in the calculations are shown in Table 52. A comparison of carbon aromaticities from the NMR analyses is also provided in this table.

Data from AFWAL/POSF.

Contracting the %H, from 100.

ND shows a value less than 0.5.

TABLE 52. CARBON AROMATICITY FROM HYDROCARBON-TYPE ANALYSIS DATA

i,								ase Spectro	metry	TAC HOR
Puel Constituents	Density,	Vol.,	We,	Carbon No. ²	Impiriosi Chemical Formula	Average Structure Assigned ^d	Nole Percent Aromatic Carbon ^e	Mole Percent Aliphatic Carbon	Carbon Aromaticity	Carbon Aromaticity
					ERAS TU	<u>rr 78</u>				
				<u> 13.30</u>						-
Paraffins Monocycloparaffins	0.75 0.81	19.1 6.8	16.6 6.4	13.3 13.3	Callen+a Callen	C13.3H28.6 C13.3H26.6	0	15.8 6.3		
Dicycloparaffina Alkylbenzenes	0.81 0.87	1.1 54.1	1.0 54.5	13.3 12.3	Calles-Calles-1	C13.3Haq.e	0 26, 6	1.0 28.0		
Indens/tetrelin Nephthalenes	0.93	4.8	5.2 16.3	12.3 12.3	CoMe-Callex-1	C12.3H16.6 C12.3H12.6	2.6 13.8	2.7 3.2		
-	_					Tota		57.0	0.43	0.54
					ERBS FUI	EL 38				
				13.03						
Paraffins Monocycloparaffins	0.75 0.81	46.8 27.5	43.4 27.5	13.0 13.0	Синаи+а Синаи	C13H20 C13H20	0	42.4 27.2		
Dicycloparaffins Alkylbenzenes	0.81 0.87	2.8 7.5	2.8 8.0	13.0 12.0	Синак-а Сена-Синак++	Casilas Casilas	0 4.1	2.8 4.1		
Indens/tetrain Naphthalenes	0.93	0.2 7.2	9.4	12.0	CoMe-Callex-1	Ctalie	4.9 8.0	4.9		
	2000		-	40.0	VIGORY -AMERICA	Tota		83.0	0.17	0.19
					ERRE FUEL S	DB-11.8				
				13.03						
Paraffins Monocycloparaffins	0.75 0.81	36.4	32.9 19.5	13.0 13.0	CxHax+a CxHax	C13H28 C13H28	0	31.8 19.3	a	
Dicycloparaffina	0.81	2,2	2.2	13.0	Синаи-а	Craffas	Ö	2.1		
Alkylbenzenes Indens/tetralin	0.87 0.93	25.1 6.8	26.3 7.6	13.0 13.0	Celle-CxHax+1 Celle-CxHax-1	C12H18 C12H18	13.3 3.9	13.4 3.9		
(Aphthalenes	1.00	9.5	11.5	12.0	C18H7-CxHax+1		10.2	<u>2,1</u> 72,6		
						Tota	18 27.4	72.6	0.27	0.35
					ERRE FUEL	3B-13·3				
• 45 10				13.03						
Paraffins Monocycloparaffins	0.75 0. 8 1	41.0 23.2	37.4 22.9	13.0 13.0	CxHax+a CxHax	C13H28 C13H26	0	36.5 22.5		
Dicyclopuraffins Alkylbenzenes	0.81 0.87	2./ 16.9	2.7 17.9	13.0	CxHgx-a CeHm-CxHex+1	Ciallas Ciallis	0 9, 2	2.6 9.1		
Indens/tetralin	0.93	7.7	8.8	12.0	CeHm-CxHax-1	Cialite	4.5	4.6		
Naphthelenes	1.00	8.5	10.3	15.0	C10H7-CxH2x+1	C12H12 Tota	9,2 18 22,9	<u>1.8</u> 77.1	0.23	0.27
						101	12.7	,,,,	0.43	0.27

aVolume percent is measured by ASTN D 2789 Nethod.

beight percent was calculated from the average density values shown in the table and the volume percents.

^CThe everage carbon number shown at the top of each column was obtained from an ASTM D2887 simulated distillation analysis. The average carbon number is the number of carbons in the normal paraffin which has the GC retention time at which 50% of the fuel is eluted. These values show excellent agreement with average carbon numbers determined as a part of the ASTM D 2425 calculation.

d. This structure was assigned hased on the carbon number. It is assumed that an aromatic compound with a boiling point equivalent to that of an sliphatic compound will contain one less carbon.

^eFor the total fuel and calculated from the structure of the average compound.

Conclusions

On the average, the mass spectrometric carbon aromaticity values were 17% lower than the corresponding NMR values. This is comparable to the 15% difference in results obtained in similar analyses on other fuels. It is worthy of note that the results from both sets of analyses are proportional to each other, and the difference in results may be due to the fact that several assumptions such as carbon number, chemical structure, and average density must be made for the mass spectrometric analyses.

Latent Heat of Vaporization

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Latent heat of vaporization (ΔH_{v}) can be routinely calculated for a pure compound using the Clausius-Clapyron equation:

$$\log \frac{P_2}{P_1} = \frac{\Delta H_V(T_2 - T_1)}{2.303 \text{ RT}_2 T_1}$$

where vapor pressures determined as a function of temperature are used for this computation. Since this approach has also been used for simple mixtures, its applicability to the calculation of ΔH_V for the subject fuels was therefore investigated. A brief examination of ΔH_V literature values for a wide range of hydrocarbons showed that most values fell within the general range of 65 to 105 calories per gram. Thus it would be expected that a fuel composed of a broad range of hydrocarbons would have a ΔH_V value well within this range. Since calculations using fuel vapor pressure data produced unreasonably low ΔH_V values (typically 30 cal/gram), it was concluded that this approach was not suitable for complex mixtures such as fuels.

Direct calorimetric measurement of this property for complex mixtures is fraught with difficulties and is thus considered impractical. Consequently, heat of vaporization was calculated using literature

ΔH, values for the various fuel components and their weight percents as determined by the hydrocarbon type analysis. A simulated distillation analysis (ASTM D 2789) was first conducted to determine the boiling range and average carbon number for each fuel. This value was used to define the compounds whose literature values should be used in the AH, calculations. Fuels, of course, are composed of a number of compound classes, each having a wide carbon number range. Fortunately, the heat of vaporization changes only gradually with increasing carbon number within an homologous series. Therefore, the $\Delta H_{_{\mathbf{U}}}$ values for compounds having the average carbon number will be representative averages for all compounds present in the homologous series of each compound class. For example, AH, values for n-hexane, n-decane, and n-tetradecane are 87.5, 86.3, and 85.7 cal/gram, respectively. The average carbon number in this example is C_{10} while the average ΔH_{\star} , value is 86.5 cal/gram, which is very close to the actual 86.3 value for n-decane. The average carbon number for the subject fuels was 13 for the aliphatic portion and 12 for the aromatic portion.

Heat of vaporization values for C_{13} aliphatic compounds and C_{12} aromatic compounds were used in computing ΔH_V values for each fuel. The average ΔH_V value for all listed isomers within a compound class was multiplied by the weight percent (Table 52) of that compound class in each fuel. The products for all compound classes were then summed to obtain the total ΔH_V for each fuel. Results are shown in Table 53.

The literature shows a ΔH_V value for only n-tridecane among the numerous C_{13} paraffins. Therefore, data for C_{10} isomers were utilized for calculating the average ΔH_V , since C_{10} and C_{13} normal alkanes have very similar ΔH_V values.

TABLE 53. CALCULATED LATENT HEAT OF VAPORIZATION FOR FUELS AT 25°C

	No. of	Avy AL, b		Puel Compos	ment Contri for Fuels	bution ⁰ and at 25°C, ca	Calculated
Puel Commonente	Tours 1.5	941/95(8	G. CAL/T	3		39-11.8	39-12.3
Cts Paraffine	374	77.9	3.3	12.9	33.9	25.7	29.2
C13 Monocycloparaffins	•	76.8	1.7	4.9	21.2	15.0	17.6
Cas Dicycloperaffine	4	81.9	2.9	0.8	2.3	1.8	2.2
C12 Alkylbenzenes	4	81.2	1.3	45.2	6.6	21.0	14.9
Coa Tetreline	12	89.3	1.3	4.6	8.4	6.8	7.8
Can Hapithalenes	4	103.9	3.9	17.0	9.3	12.0	10.7
Total	beat of va	porimation f	or fuels	85.4±2.1 [£]	81.722.6	83.1±2.3	82.4±2.4

number of isomers used in calculating everage AM, values.

It was observed that, in general, the aromatic compounds have a higher ΔH_V than similar non-aromatic compounds, and that branched paraffins have a lower ΔH_V than the corresponding normal paraffins.

Thermal Conductivity

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Thermal conductivity for the four ERBS fuels was measured by the transient hot wire method (ref. 10). In this procedure a constant heating current is applied abruptly to the resistance wire immersed in the fuel. The change in temperature of the wire following application of the current is obtained from the observed change of voltage across the wire and the known resistance-temperature characteristics.

Each fuel was analyzed 8-10 times at each of three temperatures, using four power levels and two time factors for each analysis. Thermal conductivity was then calculated in both SI and English units, with the results presented in Table 54. Also shown in this

^bAverage of isomer values found in "Physical Properties of Chemical Compounds," American Chemical Society, 1955.

Cvalues were obtained by multiplying the average component AM, by the weight percent in the fuel.

dee text for explanation of compounds selected.

^aNo values for Indans were found in the literature.

²A weighted average standard deviation.

TABLE 54. THERMAL CONDUCTIVITY OF ERBS FUELS

Measurement unit and Precision	Test temp., °C(°F)	thermal	l conduct:	and precisi ivity expre English un 3B-11.8	essed
W/m·°K ^b	0(32)	0.1277	0.1229	0.1244	0.1232
Btu/ft·hr·°F ^c		0.0738	0.0710	0.0719	0.0712
% Rel., σ ^d		0.80	0.91	0.87	0.92
W/m·°K	20(68)	0.1246	0.1190	0.1219	0.1204
Btu/ft·hr·°F		0.0720	0.0688	0.0704	0.0696
% Rel., σ		1.16	0.79	0.77	0.62
W/m·°K	40(104)	0.1221	0.1164	0.1180	0.1174
Btu/ft·hr·°F		0.0705	0.0673	0.0682	0.0678
% Rel., σ		9.77	1.52	0.74	0.72

^aAverage of 8-10 analyses per sample.

table is the percent relative standard deviation of the individual analysis results from the averages. A plot of thermal conductivity versus temperature is presented in Figure 61 for only the SI units, which currently are the preferred units of measurement. The polynominal equation constants which can be used to calculate thermal conductivity at any temperature are presented in Figure 62.

Specific Heat

Heat capacity or specific heat was measured by a standard thermo-analytical technique, using a Perkin-Elmer DSC-1 differential scanning calcrimeter and sapphire as the reference material. The fuel samples were hermotically sealed in aluminum sample pans and heated from 62 to 92°C at a rate of 10°C per minute. The amount of heat required to raise the temperature of the fuel over this

bSI unit of measurement in Watts per meter per degrees Kelvin.

CBtu/ft·hr·°F is equal to 0.5778 W/m·°K.

dPercent relative standard deviation of individual measurements from the average.

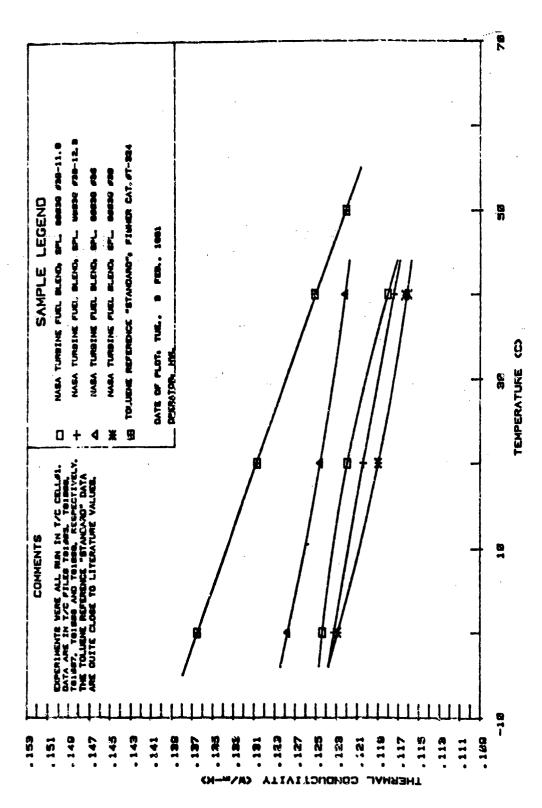


Figure 61. Thermal conductivity versus temperature.

CONSIANTS FROM CURVE FITTING DATA FROM BANPLECS)

Y = 6g + A18X > A28X8X Y = THERMAL CONDUCTIVITY X = TEMPERATURE

PCC - POLYNOHIAL CORRELATION COEFFICIENT

SAMPLI NANE 972 PCC 952 LIMITS AS AL POS AS AS POST STATES AS AS PCC 952 LIMITS	A0 ********	A1 ********	A2 ********	PCC ********	95x Limits ilteresesses
MRC/MASA TURBINE FUEL BLEND; SPL. 80039 436-11.8	.124367	0000846	0000846000001638	. 941	. 60198
MRC/WASA TURBINE FUEL BLIND; SPL. 80039 #38-12.3	.123223	0001349	000000237	.934	.00178
MRC/NASA TURHINE FUEL BLEND; SPL. 80039 #35	.127735	0001754	.00000008765	.894	. 00225
MRC/NASA TURBINE FIJEL BLEND; SPL. 80039 43B	. 122868	0002214	.00000013442	.918	.00237
TOLUEME REFERENCS "STANDARD"; FISHER CAT.#T-324	.136504		000284460088007E	.977	.00243

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Polynomial equation coefficients and constants for thermal conductivity data. Figure 62.

temperature range was compared to the heat required to raise the temperature of the sapphire external standard over the same range, and specific heat of the fuel was then calculated at 5°C intervals from the known specific heats of sapphire. Specific heat was also determined for sapphire (against a sapphire reference) and reagent grade toluene to evaluate the accuracy of the instrumentation and method. Results are shown in Table 55.

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Percent Carbon and Hydrogen

The ERBS fuel samples were submitted to two commercial analytical laboratories for duplicate carbon and hydrogen analyses. Both laboratories, Galbraith Laboratories, Inc. and Schwarzkopf Microanalytical Laboratory, use the classical combustion technique for their analytical approach. Results are shown in Table 56.

TABLE 55. SPECIFIC HEAT OF ERBS FUELS AND REFERENCE MATERIALS

	Specific Heat in cal/g°C or Btu/lb°F at Following Temperatures							
	67°C	72°C	77°C	82°C	87°C	92°C		
Sample Tested	153°F	<u>162°F</u>	<u>171°F</u>	180°F	187°F	198°F		
Fuel 35	0.446	0.447	0.446	0.450	0.455	0.459		
Fuel 3B	0.538	0.565	0.587	0.619	0.654	0.687		
Fuel 3B-11.8	0.477	0.487	0.496	0.508	0.521	0.532		
Fuel 3B-12.3	0.502	0.513	0.515	0.536	0.561	0.562		
Sapphire	0.205	0.206	0.207	0.208	0.210	0.213		
Literature values	0.204	0.206	0.208	0.210	0.212	0.214		
% Difference	0.5	0.0	-0.5	-0.9	-0.9	-0.5		
Reagent Tolueneb	0.446	0.446	0.455	0.466	0.474	0.483		
Literature values	0.436	0.440	0.444	0.449	0.455	0.461		
% Difference	2.4	1.3	2.4	3.7	4.2	4.9		

a Sapphire in the sample pan was analyzed against sapphire in the reference pan. This was a measure of the accuracy of the instrumentation.

barhis measurement reflected a combination of the instrumental accuracy and the purity of the toluene.

TABLE 56. PERCENT CARRON AND HYDROGEN IN ERBS FUELS

*** *** *			* * * * * * * * * * * * * * * * * * *				
Sample		Percent	<u>t Hydrogen Data</u>	From	Percel	nt Carbon Data	From
Number		Galbraith	Schwartskop!	Average	Galbraith	Schwartskopf	Average
38		9.97 10.09	10.20 10.41		89.65 89.58	89.53 89.42	
	Avg.	10.03	10.31	10.17	89.61	89.47	89.54
32		12.40 <u>12.35</u>	12.82 12.68		87.49 87.61	87.16 87.34	
		12.37	12.75	12.56	87.55	07.25	87.40
31-11.8		11.79 11.91	11.95 11.92		87.73 87.79	88.17 88.26	
		11.85	11.93	11.89	87.76	88.21	87.99
38-12.3		12.48 12.13	12.38 12.27		87.43 <u>87.21</u>	87.76 87.78	
		12.31	12.33	12.32	87.31	87.77	87.54

18 SULFUR ANALYSIS OF SEVEN LUBRICANT SAMPLES

Seven lubricant samples were analyzed by Schwartzkopf Microanalytical Laboratory for total sulfur. The results are shown in Table 57.

TABLE 57. SULFUR CONTENT OF LUBRICANTS

<u>number</u> <u>ppm</u> OP-234-1 6	r,
	_
	_
-2 55	6
- 3 6	3
-4 1,10	0
- 5 32	5
- 6 32	7
-7 6	2

19. MERCAPTAN SULFUR, TRACE METALS, AND PEROXIDE ANALYSES OF SHALE-DERIVED AND PETROLEUM-BASED JP-4 FUELS

Two samples each of shale-derived JP-4 and petroleum-based JP-4 were analyzed for peroxide content, mercaptan sulfur, and trace metals content. These fuel samples had been in contact with

Goodyear polyurethane bladder material during six-month compatibility studies at 140°F. The complete identification of each sample is provided below.

Sample	Labeling
81002-1	82-C-39 Volume Swells 6 months at 140°F in Shale Oil JP-4; in 9/3/80, out 3/3/81
31002-2	82-C-39 Dog Bones 6 months at 140°F in Shale Oil JP-4; in 9/3/80, out 3/3/81
81002-3	82-C-39 Volume Swells 6 months at 140°F in Petroleum JP-4; in 9/3/80, out 3/3/81
81002-4	82-C-39 Dog Bones 6 months at 140°F in Petroleum JP-4 changed every 30 days, in 9/3/80, out 3/3/81

Peroxide content was determined by ASTM Method D 1563-60, in which a standardized sodium thiosulfate solution and starch indicator are used to titrate released iodine. The method has a minimum detectability of 0.1 ppm, a repeatability of 0.4 ppm, and a reproducibility of 1.3 ppm.

Mercaptan sulfur was determined by UOP Method 163-80, which utilizes a potentiometric titration with alcoholic silver nitrate. This method has a minimum detectability of 1 ppm. It has a repeatability of 0.1 ppm at the 3 ppm level and 0.5 ppm at the 10 ppm level. Analytical results for both mercaptan sulfur and peroxide are presented in Table 58.

TABLE 58. PEROXIDE AND MERCAPTAN SULFUR IN JP-4 FUELS

JP-4 sample description	Peroxide, ppm	Mercaptan sulfur, ppm
81002-1, Shale, Volume Swell	3.2	2.9
81002-2, Shale, Dog Bones	2.6	3.3
81002-3, Petroleum, Volume Swall	2.8	3.3 ND
81002-4, Petroleum, Dog Bones	2.8	ND

aND - not detected at 1 ppm level.

Trace metals content was determined by emission spectrographic analyses. Two gram quantities of fuel were deposited into the carbon electrodes by the dropwise addition/fuel evaporation technique. This was followed by the addition of 10 milligram quantities of lithium carbonate to simulate the matrix of the 10, 100, and 1,000 ppm metal standards used in the analysis. Each standard was a mixture of about 50 different metals. Each fuel was analyzed in duplicate, and the estimated precision of the analysis was about ±100%. Average results are shown in Table 59.

TABLE 59. EMISSION SPECTROGRAPHIC ANALYSES OF JP-4 FUELS

	Approx. detection	Quantities of each metal in following fuels, ppm			
	limit,	81002-1	81002-2	81002-3	81002-4
<u>Metal</u>	ppm	Shale	Shale	Petroleum	Petroleum
Magnesium Silicon Iron Aluminum Copper Sodium Chromium Lead Tin Cadmium Zinc	0.1 0.5 0.05 0.05 0.03 0.01 0.1 0.5 0.2	0.2 0.5 0.1 0.05 0.03 0.01 ND ND ND ND	0.2 0.5 0.1 0.05 0.1 ND ND ND ND	ND a 0.5 0.05 ND ND ND ND ND ND ND ND ND ND ND ND ND	ND 0.5 ND ND ND ND ND ND ND ND ND ND ND ND ND
Nickel	0.1	ND	ND	ND	ND

^aND - not detected at indicated levels.

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20. ANALYSIS OF SHALE-DERIVED JP-4 SAMPLE NUMBER 15B FOR PEROXIDES, TRACE METALS, AND MERCAPTAN SULFUR

A sample of shale-derived JP-4 produced in the HRI refining program and returned from the GE Evendale Plant was analyzed for peroxide content, mercaptan sulfur, and trace metals content. The sample was then placed in a soft glass quart bottle covered with aluminum foil and was stored at 140°F for a total of eight weeks. Samples were withdrawn after 1, 2, 3, 4, 6, and 8 weeks for peroxide analyses.

Peroxide content was determined by ASTM Method D 1563-60, which is a titrimetric analysis utilizing a standard sodium thiosulfate solution and starch indicator. The method has a minimum detectability of 0.1 ppm, a repeatability of 0.4 ppm, and a reproducibility of 1.3 ppm.

Mercaptan sulfur was determined by UOP Method 163-80, which utilizes a potentiometric titration with alcoholic silver nitrate. This method has a minimum detectability of 1 ppm and has a repeatability of 0.1 ppm at the 3 ppm level and 0.5 ppm at the 10 ppm level. Analytical results for both mercaptan sulfur and peroxide are presented in Table 60.

Trace metals content was determined by emission spectrographic analyses. Two gram quantities of fuel were deposited into the carbon electrodes by the dropwise addition/fuel evaporation technique. This was followed by the addition of 10 milligram quantities of lithium carbonate to simulate the matrix of the 10, 100, and 1,000 ppm metal standards used in the analysis. Each standard was a mixture of about 50 different metals. Each fuel was analyzed in duplicate, and the estimated precision of the analysis was about ±100%. Average results are shown in Table 61.

TABLE 60. PEROXIDE AND MERCAPTAN SULFUR IN SHALE-DERIVED JP-4 SAMPLE 15B-792009

Time at 140°F, weeks	Peroxides,	Mercaptan sulfur, ppm
0	ND (<0.1)	4.3
1	1,1	
2	1.1	
2	0.9	
4	0.9	
6	1.2	
8	0.8	

TABLE 61. EMISSION SPECTROGRAPHIC ANALYSIS OF SHALE-DERIVED JP-4 SAMPLE 15B-792009

Metal	Amount detected (±100%), ppm
Magnesium	0.2
Silicon	2
Iron	0.1
Aluminum	3
Copper	0.03
Sodium	0.5

21. EXAMINATION OF SHALE-DERIVED JP-4 FOR COMPONENTS CAUSING ELASTOMER DETERIORATION

Introduction and Summary

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A specimen of shale-derived JP-4 was found to exhibit compatibility problems with elastomeric fuel system components and bladder material during Air Force tests. The observed elastomer deterioration was initially suspected to occur because of peroxide compounds present in the fuel. Peroxide numbers, however, were determined for the fuel specimen in question, as well as for a number of related fuels, with the result that no significant peroxide levels were found.

In an effort to identify problem-causing fuel components, comparative analyses were conducted on both the problem fuels and those that presented no problems. The offending component(s) were suspected to be sulfur compounds. Analyses were, therefore, conducted by gas chromatography using a sulfur-specific Hall electrolytic conductivity detector. Additionally, wide-scan GC/MS analyses were conducted on the fuels, and fuel concentrates were prepared and analyzed. All of these analyses and their significance are discussed in the following paragraphs.

Peroxide Analysis

Peroxide numbers were measured for a total of fifteen different fuels using ASTM Method D 1563-60. In this method, the sample is diluted with carbon tetrachloride and placed in contact with an aqueous potassium iodide solution. Peroxides present are reduced by the potassium iodide with a stoichiometric release of iodine. The iodine is titrated with a standard sodium thiosulfate solution using starch as the end-point indicator. The method has a minimum detectability of 0.1 ppm, a repeatability of 0.4 ppm and reproducibility of 1.3 ppm. Results of the analyses are presented in Table 62.

In no case was the peroxide level sufficient to cause the observed elastomer deterioration.

Sulfur-Specific Gas Chromatographic Analyses

The problem component(s) of the fuel were believed to be sulfur compounds. The fuels were thus screened for the presence of sulfur-containing components using a gas chromatograph equipped with a sulfur specific detector. A Tracor Model 560 gas chromatograph with a 700A Hall electrolytic conductivity detector (HECD) and a 12-meter methylsilicone fused silica capillary column was used for these analyses. The 700A HECD, when operated in the

TABLE 62. PEROXIDE ANALYSES OF SHALE-DERIVED AND PETROLEUM-BASED JP-4 FUEL

Sample number	Description and history of fuel	Peroxide,
	Shale Oil JP-4 tested 6 months at 140°F; in 8/20/80, out 2/20/81	
1 2 3	Goodyear 51956 seam adhesion Pliocel seam adhesion FT-136 repair adhesive	0.8 2.3 0.9
	Petroleum JP-4 (changed every 30 days) tested 6 months at 140°F; in 8/20/80, out 2/20/81	
4 5 6	Goodyear 51956 seam adhesion Pliocel seam adhesion Repair adhesive	1.4 1.9 1.7
	Shale Oil JP-4 tested 6 months at 140°F; in 9/3/80, out 3/31/81	
7 8	82C 39-A 82C 39-B	2.7 2.8
	Petroleum JP-4 (changed every 30 days) tested 6 months at 140°F; in 9/3/80, out 3/3/81	
9 10	82C 39-A 82C 39-B	2.0 2.5
11	Shale JP-4 from UDRI, John Dues to Charles Martel, 3/3/81	3.3
	Shale oil JP-4 fuels from Request 80007 and Report 80-18	
12 13	HRI-LO-2054 (80-PHJ-084A) HRI-LO-2057 (80-PHJ-084B)	1.8 0.4
14	Pratt and Whitney Shale Oil JP-4, Sample ES 0001A, Previously analyzed: Request	ND ^a (<0.1)
15	80022, Report 80-31 Petroleum JP-4, sample GEC-145-400-792033, Previously analyzed: Request 79030, Report 79-45	2.0

sulfur mode, is highly selective for sulfur compounds, but also shows a small response to high levels of hydrocarbons. This small signal obtained for hydrocarbons was found to be useful in establishing the relative retention characteristics of detected compounds.

Gas chromatographic conditions used were:

Injection Port Temperature: 200°C

Detector Base Temperature: 225°C

Reactor Temperature: 850°C

Carrier: Helium

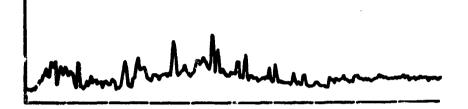
Initial Column Temperature: 60°C for 4 min

Temperature Program Rate: 10°C/min Final Column Temperature: 200°C

Several petroleum JP-4 fuels were included in the analyses in addition to various specimens of the shale fuel. A number of the shale fuel samples exhibited a sulfur component eluting at approximately 19 minutes. In no case was this component detected in petroleum JP-4. The fuel coded HRI-LO-2057 had the highest concentration of the sulfur compound (Figure 63-a). That fuel also contained a second sulfur component having a retention time of approximately 1.5 minutes. This retention time is close to that of dimethyl disulfide. No sulfur-containing compounds were detected in the shale JP-4 HRI-LO-2054 (Figure 63-b). The results of the evaluation are summarized in Table 63.

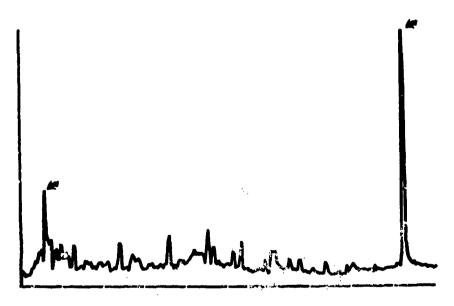
A number of reference blends were prepared and analyzed to determine whether standard fuel additives, or benzotriazol which is known to have been added to some lots of the fuel, are related to the sulfur-containing component. The standard additives used for test blends were DCI-4A corrosion inhibitor and the major components of ethyl antioxidant 733 (2,6-ditert-butylphenol and 2,4,6-tritert-butylphenol). Dimethyl disulfide was used in the

SAMPLE: HRI-LD 2054 HLJECTED AT 14:39:51 OH MAR 27: 1901 METHOD: HALLS RAW HS14:J5 PRC: #PRC30 ENLARGE= 25 IF2: 0 TO 20 MIN 1 DIV= 2 MIN:



(a) Fuel HRI-LO-2054-no sulfur components.

SAMPLE: HRI-LO 2057 INJECTED AT 14:10:27 ON MAR 27, 1901 METHOD: HOLLS RAM: HS13:J5 PRC: #PRC30 ENLANCE: 1 TIME: 8 TO 25 MIN: 1 DIU: 2 MIN)



(b) Fuel HRI-LO-2057-sulfur components marked with arrows.

Figure 63. Gas chromatograms of Shale 3P-4 using Hall electrolytic conductivity detector in subject mode.

TABLE 63. RELATIVE AMOUNT OF SULFUR COMPONENT IN FUELS TESTED

Relative Intensity of Peak for Sulfur Com-Sample No. and/or Description ponent Compared to Same Peak in Fuel HRI-IG-2057

	20 iiot min, on popularitania politica compared no	Domic Louit III
	h	
	HRI-LO-2057 ^b	100
	JP-4 15B	30
	ES0001A	_c
	UDRI J2-4	15
5.	HRI-LO-2054	-
6.	JP-4 79003	-
7.	JP-4-14-70	-
8.	Shale Oil 82C39A	- 25
9.	Petroleum 82C39A	-
10.	x190-64	15
11.	x190-65	35
12.	x190-66	25
Rl	3.8 ppm Benzotriazole in EOTH	-
R2	3.8 ppm Benzotriazole in Shale Oil JP-4	30
R3	3.8 ppm Benzotriazole in Petroleum JP-4	-
R4	11 ppm DCI-4A plus 3 ppm (CH ₃) ₂ S ₂ in Shale Oil	31
	JP-4	
R5	11 ppm DCI-4A plus 3 ppm (CH ₃) ₂ S ₂ in Petroleum	•
i	JP-4	
R6	31.6 ppm antioxidant plus 3 ppm (CH ₃) ₂ S ₂ in	28
	Shale Oil JP-4	
R7	31.6 ppm antioxidant plus 3 ppm (CH ₃) ₂ S ₂	-
	petroleum JP-4	
R8 -	3.2 ppm benzothiazole in Shale Oil JP-4.	30
R9	3.2 ppm benzothiazole in petroleum JP-4	-
	LO-2057 N ₂ evaporated (spl. #1)	330
	LO-0057 after further evaporation of volatiles	1180
JP-4	15B pot residue after distillation at	-
	almospheric pressure	
JP-4	15B tenth fraction from atmospheric	-
	pressure distillation	
JP-4	15B pot residue solids dissolved in pentane	-

a Component with \sim 18 min. retention time, near C₁₈ hydrocarbon

b Also contains sulfur component with '1.5 min. retention time.

C Dash = non detected

fuel refining process, thus this compound was also added to determine any reactive effects. The blends were prepared using the shale JP-4 coded 15B and a petroleum JP-4. Benzothiozol was included among the additives evaluated because it is sometimes used as a corrosion inhibitor and antioxidant, and it has a name easily confused with benzotriazol. From these analyses, which are included in Table 63, it can be concluded that the presence of the various additives has no effect on the magnitude of the sulfurcomponent peak.

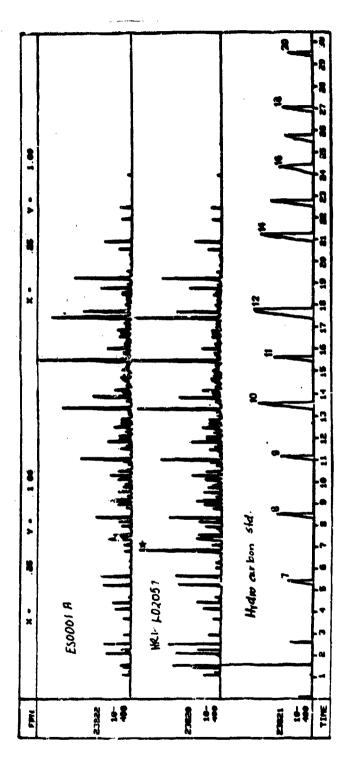
Gas Chromatography/Mass Spectrometric Analyses (GC/MS)

In order to identify the sulfur component found in some of the shale JP-4 specimens, GC/MS analyses were conducted on two fuels, one containing the unidentified component (HRI-LO-2057) and the other being free of that component (ES 0001A).

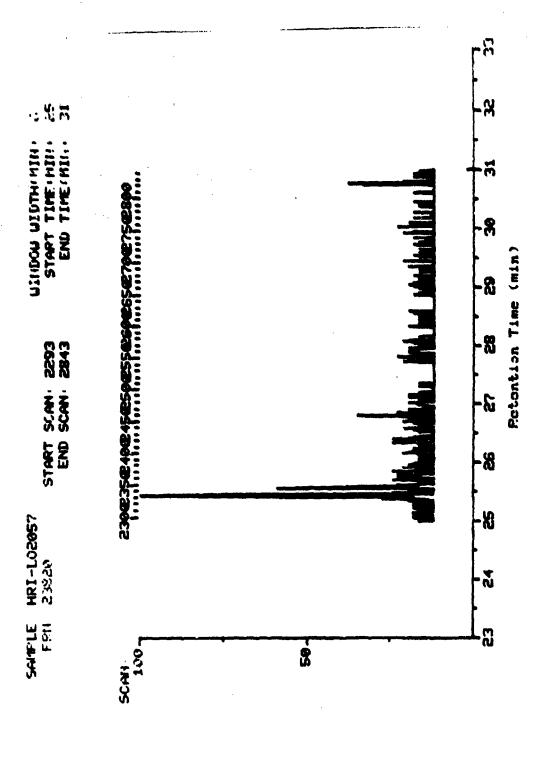
A 25-meter OV-101 fused silica capillary column was used for the separation. The unidentified component is known to fall in the boiling range of C₁₈ paraffins. A hydrocarbon reference mixture was chromatographed under the same conditions as the samples in order to determine the expected location of the unknown peak. Figure 64 presents a composite of the three reconstructed total ion chromatograms. Hard copy mass spectra were obtained for each component shown in the chromatogram. The only difference found between the two fuels lies in the concentration of toluene which is substantially higher in HRI-LO-2057. That peak is marked with a star in Figure 64.

Based on the hydrocarbon reference, the sulfur component has an expected retention time of 26-28 minutes. In order to further examine the HRI-LO-2057 data for components in this area of the chromatogram, the scale was greatly expanded through a six minute retention time window from 25 to 31 minutes as shown in Figure 65. The only components detected were C_{17} paraffins at approximately

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Reconstructed total ion chromatogram of a good shale fuel (ES-0001A) and problem fuel HRI-LO-2057 compared to hydrocarbon standard mixture. Figure 64.



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Examination of reconstructed total ion chromatogram of fuel HRI-LO-2057 - expanded scale, six minute window. Figure 65.

25.5 minutes. No other peaks were detected, with the detection limit being defined as three times baseline noise.

Concentration of Sulfur Component in Fuel

In order to concentrate the sulfur component in the fuel, a simple one-plate distillation was performed in which the fuel was separated into 10 fractions plus a small residue. The component sought was expected to be in the last fraction or in the residue. Analysis of the fraction by GC, however, showed that the sulfur component did not appear in any of the fractions or in the residue. The compound was apparently destroyed during distillation.

Several other concentration techniques were also employed. An aliquot of fuel was passed through a silica gel column where the sulfur compound was retained. The component was recovered from the gel by elution with a 50/50 chloroform-ethanol mixture. The level of the component in the solvent was approximately double its concentration in the original fuel. It was, however, readily concentrated further by evaporation of the excess solvent.

Simple evaporation of the original fuel under a nitrogen flow was also found to be a means by which a 10-fold increase in the concentration of the sulfur compound could be achieved.

Vacuum distillation was also used to obtain a more concentrated specimen of the sulfur compound. Distillation data are presented in Table 64. The component was found in the residue after vacuum distillation.

Analysis of Concentrates

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GC/MS analyses were conducted on several of the concentrates. A reconstructed total-ion chromatogram of the concentrate obtained by evaporation of the fuel volatiles is presented in Figure 66.

TABLE 64. VACUUM DISTILLATION DATA FOR FUEL 15B

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Fraction number	Boiling range,
1	29-45
2	45-60
3	60-75_
4	40-48 ^a
5	48-58
6	58-73
7	73-81
8	81-90
Residue	90

aPressure decreased.

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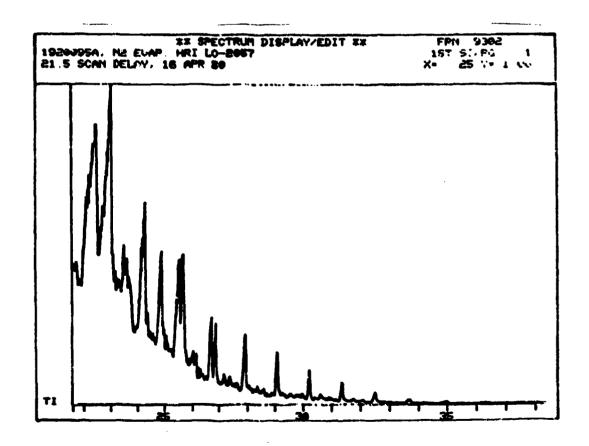


Figure 66. Reconstructed total ion chromatogram of sample HRI-LO-2057 after evaporation of volatiles.

This concentration procedure yields a sample similar to that obtained by vacuum distillation. Mass spectra of the peaks indicate that they are due to a series of paraffin hydrorarbons. No sulfur-containing or non-hydrocarbon component was detected.

A reconstructed total-ion chromatogram for the silica gel concentrate was recorded as shown in Figure 67. The portion of the chromatogram between 25 and 30 minutes was expanded as shown in Figure 68. Three components were detected as follows:

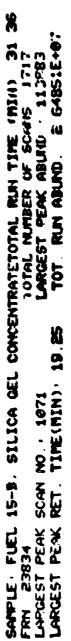
Retention time, min	Compound detected	
25.9	Phenanthrene	
26.6	2-Phenylhenzimidazol	
27. 4	Methylphenanthrene	

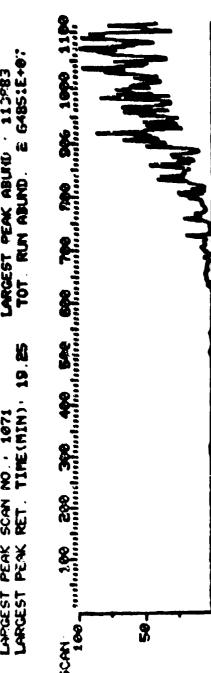
The background was examined for evidence of the sulfur compound but none was found.

Discussion and Conclusion

The sulfur compound that was detected in a number of samples using the Hall ECD could not be confirmed or identified by GC/MS, presumably because of the instability (or high reactivity) of the component. High reactivity is, of course, expected of compounds which attack elastomers (e.g., peroxides). The other experiments demonstrated the instability of the sulfur component. Not only was the component lost during distillation at atmospheric pressure, but when attempts to evaporate fuel volatiles were carried out too vigorously (by heating) the concentration of the component was found to decrease instead of increase.

The sulfur compound was found in fuels known to be problem-causing and was absent in fuels known to be problem-free, i.e., petroleum JP-4 and certain lots of shale JP-4. Due to the apparent instability of this component, the relative amounts found may not





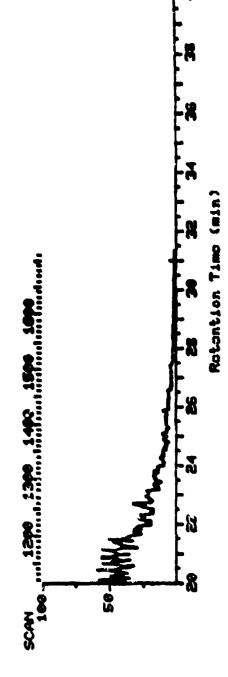
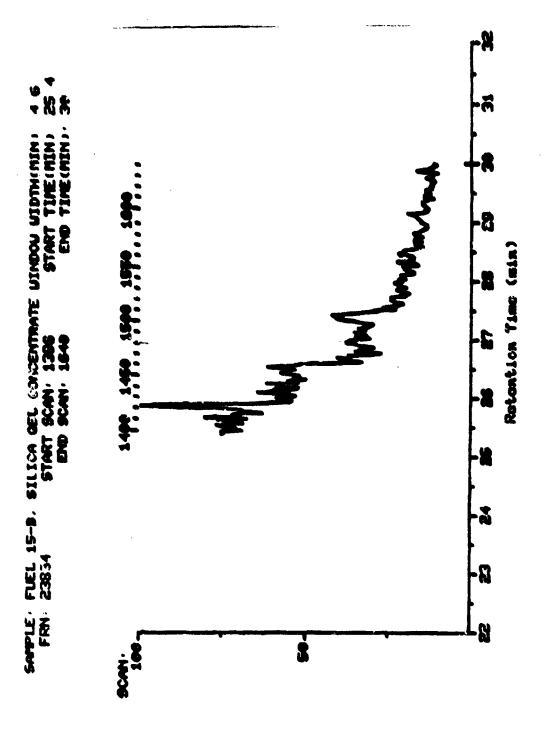


Figure 67. Reconstructed total ion chromatogram of silica gel concentrate from fuel 15B.



Portion of reconstructed total ion chromatogram of silica gel concentrate from fuel 15B. Figure 68.

have correlated in every case with the degree of polymer degradation. The component appeared to be unrelated to any of the normal fuel additives, or to the benzotriazol added to improve copperstrip corrosion properties.

The immediate goal of this investigation was to find differences between the good fuel and the problem fuel. Elastomer deterioration testing was not a part of the study, and differences found could not be conclusively related to elastomer deterioration. In order to directly relate the fuel or any of its fractions to elastomer attack, a small-scale accelerated test for elastomer deterioration would have been required.

22. ANALYSIS OF THREE REFERENCE GAS BLENDS FOR CARBON MONOXIDE CONCENTRATION

APL personnel had reason to suspect that three calibration gas cylinders of carbon monoxide in nitrogen did not contain the concentrations of CO specified in the containers. Each cylinder of gas was, therefore, analyzed for CO content by a method described in EPA Method 25. This method utilizes the TGNMO (total gaseous non-methane organics) apparatus which oxidizes the CO to CO_2 , reduces the CO_2 to methane, and then measures the amount of methane by means of a flame ionization detector. The TGNMO apparatus had been calibrated for the range of 50-10,000 ppm CO, using standards of nominally 50, 500, and 10,000 ppm.

The calibration curve for the day on which the CO cylinders were analyzed was within 3% of the validated curve (the method specifies 5%). However, the CO cylinders were analyzed directly against the 51.1 ppm CO standard since the specified CO values were so close to this standard. Both the samples and the standard were injected four times each to improve on the accuracy of the results. Data are presented in Table 65.

TABLE 65. CARBON MONOXIDE IN NITROGEN

	51.1 ppm CO CO in air	Three cylinders of CO in nitrogen		
	air standard		Tank 2	
Detector response	64,842	107,871	42,247	73,356
in area counts for	62,767	110,553	41,807	76,898
four replicate	64,794	110,258	42,541	76,867
sample injections	63,434	110,608	42,797	76,869
Mean counts	63,960	109,823	42,348	76,123
Standard deviation, G	1,028	1,310	425	1,511
Percent relative, o	±1.6	±1.2	±1.0	±2.0
Determined CO in N2, ppm		87.7	33.8	60.8
Value on label, ppm		87	37	66

23. TRACE METALS ANALYSIS OF THERMALLY UNSTABLE JP-4 SAMPLES

Five samples of JP-4 coded YAH-64 were analyzed for trace metals using an ISA Model JY48P inductively coupled plasma (ICP) spectrometer. The purpose of the analyses was to determine the source of the thermal instability problem. The ICP analyses were conducted on aqueous acid extracts (ultrapure HCl) of the fuels which provided a concentration factor of 22.5 for the metals. The test results for an acid extraction blank, the fuel samples, and a reference standard are shown in Table 66.

With the exception of 183 ppb zinc in one of the fuel samples (T-I), none of the metal concentrations appears high enough to cause a thermal stability problem.

24. IDENTIFICATION OF SULFUR CONTAINING CONTAMINANT IN SHALE JP-4

A specimen of shale-derived JP-4, coded VN-81-149, was found during its routine chromatographic analysis within the Aero Propulsion Laboratory to contain an unusually high boiling component. The component, which represented approximately 0.2% of the sample, had a boiling point between those of normal C_{21} and C_{22} paraffins.

TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA SPECTROMETRY TABLE 66.

(qua) (pap)	9 Becovery	111	108	16	112	300	111	•	Z	103	105	1	100	103	911	*	•
itandard in 39	True Value	330	3	92	165	360	235	•	135	315	315	•	3	430	120	335	•
Reference S	Observed	38	3	182	185	358	262	•	127	326	332	•	8 2	#	132	329	1
qds .	긻	8.3	2	7.1	ð	£	2	2	21.8	Ş	6.0	2	2	21.7	2	182.7	₽
Fuels	1-8	6.0	₽	12.5	2	6.3	7.0	ğ	28.6	2	R	Fi	2	7.1	8.1	4.4	£
of JP-4	1-d	15.9	₽	13.4	₽	5.9	욡	£	26.3	8	1.2	2	ğ	4.0	2	21.2	Q
Extracts	1-1	26.7	2.2	7.2	2	≘	2	2	26.3	ğ	6.0	2	Ş	2	2	17.2	£
Acid Acid Extracts of JP-4 Fuels', pp	1-1	26.0	2	5.2	2	R	ð	9	14.9		1.3	₽	ē	<u>Q</u>	₽	11.9	9
HC1 Acid	Blank, pob	J _Q	줥	1.4	2	2	0.5	2	Q	皇	皇	욮	Ð	윤	₽	Q	Q
Detection	Limit, pob	**	9.0	6.0	3.2	2.5	4.0	6.7	10.0	1.6	0.2	3.3	0.1	3.1	4.3	3.3	11.1
	Element	Altminum	Cadmium	Iron	Mickel	Poron	Cobalt	Magnes tun	Lead	Chromium	Manganese	Vanadium	Beryllium	Copper	Molybdenum	Zinc	Tin

Lowest quantifiable concentration in the fuels.

b Values represent levels in original JP-4 fuel samples.

^C MD means not detected at stated limit.

Analyses were conducted to identify the component which was believed to be a contaminant of the fuel.

GC/MS was the analytical technique of choice for this identification. Figure 69 illustrates a total ion chromatogram of a portion of the fuel and of a hydrocarbon standard. Deuterated (d_{10}) anthracene which has a retention time of about 20.2 minutes, was added to both the fuel and the standard hydrocarbon mixture. fuel component to be identified had a retention time of 25.5 minutes and eluted just after the C21 n-paraffin. The mass spectrum of the unknown component indicated the presence of two atoms of sulfur in the molecule and showed that the molecule could readily cleave to give two identical fragments. The molecular weight was established as 290. A search of all available mass spectral libraries produced no match for the mass spectrum. However, based on information from the mass spectrum, a di-Ca alkyldisulfide was proposed as the general compound type with the di-n-octyl being the most likely. The presence of aulfur was confirmed by x-ray emission analysis.

Combined gas chromatography/Fourier transform infrared spectroscopy was used to assist in the identification. The component in question was concentrated by evaporation of the more volatile portion of the fuel in a stream of dry nitrogen. The infrared spectrum obtained for the component established with certainty that the compound was non-aromatic and the data supported the proposed structure, though it did not conclusively establish the identity of the compound.

The compound di-n-octyldisulfide was not readily available from regular chemical supply houses such as Aldrich, Sigma, Alfa, etc., but was finally procured through a listing of rare and fine chemicals (K&K Life Science Div. of ICN, Inc.). A mass spectrum of di-n-octyldisulfide was recorded and was found to exactly match the spectrum obtained for the fuel components as shown in

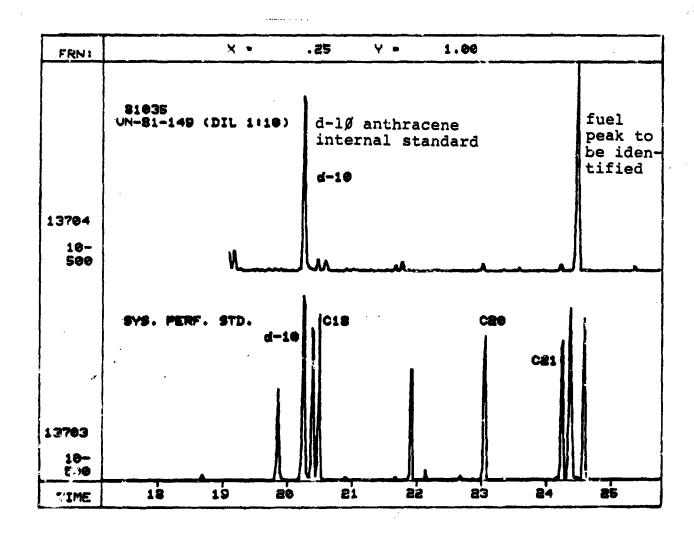


Figure 69. Reconstructed total ion chromatogram from GC/MS analysis. The fuel component is compared to a standard hydrocarbon mixture.

Figure 70. A comparison of the FTIR spectra of the fuel component and reference materials are shown in Figure 71. The fuel component has thus been unequivocally identified as di-n-octyldisulfide.

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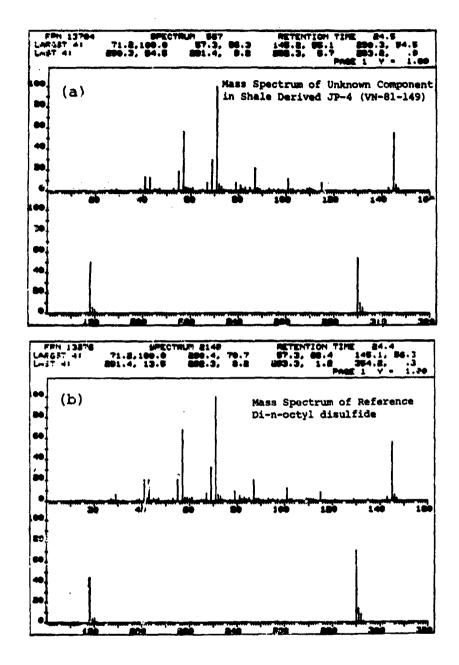


Figure 70. Mass spectra of, (a) unknown compound in shale JP-4 and (b) reference compound di-n-octyldisulfide.

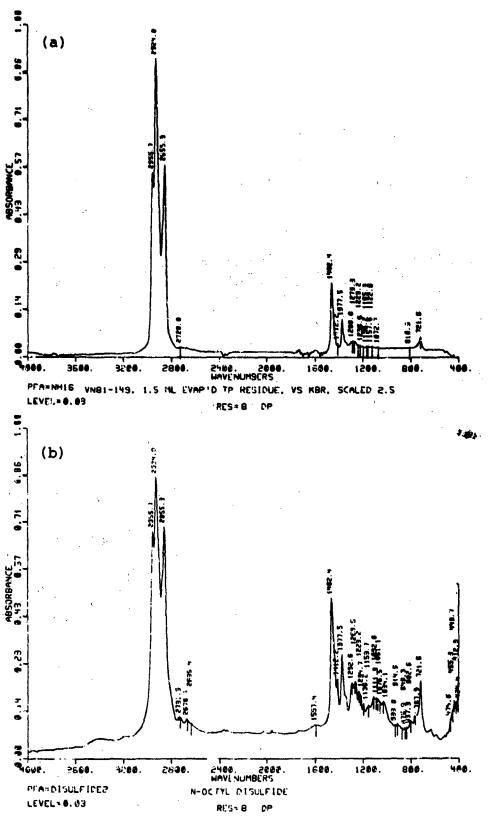


Figure 71. FTIR spectra: (a) contaminant in shale-derived JP-4, (b) reference compound, di-n-octyldisulfide.

25. OPERATION AND EVALUATION OF CARLO ERBA CHN/O ELEMENTAL ANALYZER

The capability for performing accurate and reliable elemental analyses of fuel samples is required in support of current and planned APL fuel development programs. Because of the anticipated large number of samples to test, it was considered advantageous to APL to develop this capability in-house. A Model 1104 Carlo Erba CHN/O elemental analyzer was available within the Aero Propulsion Laboratory facilities for this purpose.

The Model 1104 Carlo Erba analyzer was purchased in 1974 and had been periodically operated by Air Force personnel during subsequent years. The data from these intermittent analyses usually indicated that unreliable results were being obtained. The goals of this program were to determine why reliable data were not being obtained and then to make the analyzer functional for routine analyses of hydrocarbon fuels.

Carlo Erba Operating Procedure

Up to 23 discrete weighed samples loaded in metal containers are placed in the revolving magazine of the automatic sampler. The sampler is placed on top of a reactor where samples are introduced step-by-step and processed. Sample combustion is carried out in one of two independently heated reactors, each one linked to a chromatographic column connected in parallel to the same thermal conductivity detector.

One reactor performs the oxidation-reduction process to determine carbon, hydrogen, and nitrogen. The sample is instantaneously pyrolyzed at about 1,150°C and is then simultaneously oxidized by the combination of introduced oxygen and the action of a chromate catalyst to produce CO_2 , H_2O , and nitrogen oxides. The combustion products then pass through the copper reduction reactor at about

700°C where nitrogen oxides are reduced to nitrogen gas and the excess combustion oxygen is irreversibly absorbed. The final products pass through a Porapak Q analytical column which produces a three-peak gas chromatogram of CO₂, H₂O, and N₂.

A second reactor with its own gas chromatographic column is used for the oxygen determination. The only requirements are that another magazine be loaded with up to 23 samples and the automatic sampler be switched to the oxygen side. The sample s pyrolyzed at 1,150°C and any oxygen present is converted to CO which is then separated from nitrogen, if present, by a molecular sieve analytical column.

A CHN analysis requires 10 minutes per sample. The oxygen analysis requires 5 minutes per analysis. The Model 1120 electronic integration is recommended for determining the peak area at an accuracy of ±0.1%. An overall analytical accuracy of at least ±0.3% is claimed for the Carlo Erba Analyzer.

Instrument Set-up

A fresh cylinder of 99.99% pure oxygen gas was procured and the Carlo Erba Model 1104 was set up according to specifications for measuring C, H, and N. The oxygen side of the analyzer was not evaluated. The recommended integrator was not available so the recorder was initially connected in parallel to both a Columbia Scientific Supergrator II and a Minigrator. When it became apparent that both integrators were producing similar results, only the Minigrator was subsequently used for measuring peak areas. Upon initial start up, there was a significant amount of baseline noise in the recorder. This was found to be due to a loose wire. After this was repaired, other problems continued to appear and Mr. E. M. Becker, who is the United States representative for Carlo Erba Company (located in Italy), was contacted for guidance in solving these problems. The instrument was leak-checked after

each problem was investigated, and several times the oxidation and reduction tubes were repacked with fresh catalysts in attempts to remedy persistent problems. After making all instrument modifications and repairs suggested by Mr. Becker, the accuracy and precision of the resulting test data were still not up to specifications. This is illustrated by subsequent data from replicate injections of the acetanilide standard and actual analyses of toluene, JP-7 fuel, and cyclohexanone-2,4-dinitrophenyl-hydrazone against the standard.

Precision for Multiple Injections of the Acetanilide Standard

A series of standards was injected into the Carlo Erba analyzer after each major or minor modification of the instrument. The results of most of these injections are reproduced in Table 67 which contains k-factor values for N_2 , CO_2 , and H_2O . The k-factor is a normalized peak area per unit sample weight. The results showed that the instrument repeatability varied from day to day and in general was not very consistent.

Analysis of Samples and a Comparison With Data From Other Laboratories

Several other samples also were analyzed during the latter part of the evaluation period for the Carlo Erba instrument. These samples included two reference materials, toluene and cyclohexanone-2,4-dinitrophenylhydrazone, plus JP-7 jet fuel. The test results are shown in Table 68. As can be observed, the accuracy was not near the specified ±0.3% value, for any of the elements.

A sample of JP-7 jet fuel was then taken to an alternate cooperating laboratory for elemental analysis on a Carlo Erba Model 1106 analyzer. This model is a newer instrument than the Model 1104. One obvious difference between the two is that the liquid samples are maintained under pressure in the Model 1106 while awaiting the automatic injections from the sample magazine.

•							•									
		TABLE	67	. PRI	PRECISION	ON FOR		REPLICATE		ACETANILIDE		NJEC	INJECTIONS	10		
		11-19-80	12-4-80	1-0-61	Replicat 1-8-81 1-13-81 1-14	icate k-f [-14-8]	te k-factors and calculated pracision on the following dates 1-81 1-21-61 1-22-81 1-28-81 1-29-81 1-30-81 2-2-81 2-4-81	calculat	ed precis -28-81 1	100 on t	- follow -30-81	Ing date:	11	2-9-81 2	2-24-81	2-25-61
	Mitrogen k-factors x 10 ⁴	2.720 2.778 2.354	l .	2.257 2.237 2.337 2.340 2.373	2.39 2.39 2.39 2.29 2.29	2.23 2.28 2.22 2.22 2.23	2.13 2.12 2.09 2.06 2.16	2.21 2.25 2.25 2.32 2.12	2.13 2.12 2.09	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	2.53 2.53 2.53	2.2.2 2.5.3		######################################	2.2 2.4 2.4 2.5 2.5	4444 4444
				2.391	2.22	2.25 2.25 2.26 2.16		2.3					2.35	2.2		
	Mean Percent relative, o	2.617	2.807 10.1	2 336 2.1	2.28	2.197	2.112	2.24	2.11	2.482	2.527	2.52	2.39	2.71 1.5	2.5 5.0	2.43
187	Carbon k-factors x 10 ⁸	1.935 2.360 3.557	7.596 8.202 7.856 8.477	2.7.94 2.7.94 3.12 3.13 3.13 3.13 3.13	2.5.5.5.5.5.5.5.5.5.5.5.5.5.5.5.5.5.5.5	7.59 7.60 7.60 7.60 7.61 7.70 7.47	7.52 7.66 7.32 7.33	8.00 7.90 7.90 8.12 7.90 7.90 8.16	7.18	6.6.6.6.6.6.6.7.7.7.7.7.7.7.7.7.7.7.7.7	11.00 11.00	2. 2. 4. 2. 4. 4. 4. 4. 4. 4. 4. 4. 4. 4. 4. 4. 4.	8	8.80 7.72 7.72 7.92 7.92 7.92 7.92	1 4 8 8 1 4 8 8	9.90
	Mean Percent relative, o	2.617 32.1	6.033 4.6	8.174	7.81	7.61	7.506	6.11 1.9	7.43	8.658 0.7	9.207	6.74 4.3	9.28 2.0	7.71	9.11	# · · · ·
	Mydrogen k-fectors x 10 [§]	5.172 2.360 3.557	3.479 3.197 3.216 2.705 3.058	3.13 2.21 2.26 2.44 3.16 3.26 3.26 3.26 3.26	3.78 3.18 3.18 3.17 3.26 4.19 2.98 3.75	3.01 3.37 3.14 3.14 3.13 3.26 2.77 2.50	2.71 2.91 2.57 3.02	3.17 2.69 2.90 2.90 3.00 3.17	2.71 2.91 7.77	3.11 3.26 3.13 3.13 3.16	8.5.6. 50.2.	4.00 3.15 3.81	3.56 5.02 5.03 5.03 5.03 5.03 5.03 5.03	2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.	3.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2	3.10 3.15 3.15 3.16
	Mean Percent relative, G	3.700	3.131	3.301 5.1	3.46	3.056 8.6	2.796 6.3	2.95 5.8	2.80	3.13	3.46	3.65	3.74	3.09	3.32	3.13 0.9

		•	Perc	.51	ogen	닀	Percent cal	carbon	Per	Percent hydrogen	drogen
	Date	Sample	Theory	Found	Deviation	Theory	Found	Deviation	Theory	Found	Deviation
	1-20-81	Toluene	0.0	0.0 0.0	0.0	91.25	58.50 52.40	-32.75 -38.85	8.75	5.38	-3.37
	1-21-81	Toluene	0.0	0.0	0.0	91.25	90.4	-0.9 +2.1	8.75	16.4 16.6	+7.6 +7.8
	1-21-81	JP-7 fuel	0.0	0.0	0.0	~85.1	79.4 79.9	-5.7	14.9	13.6	-1.3
	1-28-81	JP-7	0.0	0.0	0.00	~85.1	99.3 79.2 79.8	+14.2 -5.9 -5.3	~14.9	16.2 13.8 12.6	+1.3 -1.1 -2.3
188	2-2-81	JP-7	0.0	0 0 0	0.00	∿85.1	92.7 86.7 84.6	+7.6 +1.6 -0.5	∿14.9	14.3 16.9 14.1	-0.6 -0.5 -0.8
	2-4-81	CDNH a	20.14	20.3	+0.2	51.79	51.1 50.1	-0.7	5.07	5.1	0.0
	2-5-81	CDNH a	20.14	17.5 19.5	-2.6 -0.6	51.79	46.3 81.5	-5.5	5.07	3.2	-1.9
	2-5-81	JP-7 fuel	0.0	0.1	0 1 1 1 1 1	~85.1	91.0 81.8 130.9	+5.9 -3.3 +44.8	∿14.9	14.3 15.8 17.3	-0.6 +0.9 +2.4
	2-23-81	JP-7 fuel	0.0	0.0	0.0	√85.1	88.6 89.0	+3.5 +3.9	∿14.9	15.8 16.5	+0.9

 a CDNH = cyclohexanone-2,4-dinitrophenylhydrazon= ($C_{12}H_{14}O_{4}N_{4}$).

The test results from this instrument over a two-day period are shown in Table 69. Also shown in Table 69 are test results for reference chemicals analyzed at Carlo Erba Company using a Model 1104 instrument.

Conclusions and Recommendations

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After making all instrument modifications and adjustments recommended by E. M. Becker and still not improving on the poor accuracy and repeatability of the Carlo Erba analyzer, it was concluded that the problem must be in the instrument electronics. As a minimum solution, it appeared that the instrument needed to be completely reworked by a qualified representative from Carlo Erba Company. If the analyzer could not be repaired on site, high costs probably would have made sending the unit back to Carlo Erba Company in Italy for repair an impractical alternative.

26. DETERMINATION OF AROMATIC CARBON IN TWO FUELS BY 13C FTNMR

Values for carbon aromaticity were determined on two fuel specimens by carbon-13 NMR using a Varian CFT-20 Fourier transform spectrometer. That instrument utilizes a Varian 602L computer for data acquisition, data reduction, and system control. The spectrometer was operated at 20 megahertz and the following instrument conditions were utilized for the analyses:

Sample probe	8 mm
Sweep width	4,000 Hz
Number of transients	1,000
Acquisition time	1.023 s
Pulse width 90°	17 µ/s
Pulse delay	5 s
Data points	8,192

COMPARATIVE DATA FROM A MODEL 1104 AT CARLO ERBA COMPANY AND A MODEL 1106 AT ALTERNATE LABORATORY TABLE 69.

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Analyzing organization		rerc	rcent nitrogen	oden	ĭ	Percent carbon	arbon	Ž	Percent nyarogen	irogen
				(Deviation from			(Deviation from			(Deviation from
	Sample	Theory	Found	mean)	Theory Found	Found	mean)	Theory	Found	mean)
Carlo Erba Benz	zoic acid	0.0	0.0	0.0	68,85	68.91	+0.10	4.95	4.98	+0.12
H ² C	$C_7H_6O_2$		0.0	0.0		68.55	-0.26		4.77	-0°0ن
			္ပါ	0.0		68.98	+0.17		4.83	-0.03
		mean	1	1		68.81	0.18 ^D		4.86	0.08
Carlo Erba CDNH ^a	на	20.14	20.25	+0.13	51.79	51.48	-0.24	5.07	4.95	-0.18
			19.96	-0.16		52.03	+0.31		5.15	+0.62
			20.15	+0.03		51.65	-0.07		2.30	+0.17
		nean	20.12	0.11 ^D		51.72	0.21 ^D		5.13	0.12
Alte, nate										
Laboratory JP-7 fuel	7 fuel		0.19	+0.03		86.14	+0.14		15.10	0.00
•			0.14	-0.02		86.07	₹0.07		15.10	0.00
			0.13	-0.03		86.15	+0.15		15.09	-0.01
			0.16	٥٠.٥		85.53	-0.07		15.05	-0.05
			0.11	-0.05		85.70	0.30		15.23	+0.13
			0.25	+0.03		86.08	+0.08		15.05	+0.05
			0.17	+0.01		85.90	-0.10		15.11	+0.01
			0.15	-0 01		86.72	+0.12		15.10	9.00 0
		mean	0.16	0.03 ^b		86.08	0.13 ^D		15.10	0.03 ^b

aCDNH = cyclohexanone-2,4-dinitrophenylhydrazone (C12H14O4N4).

bComputed without regard to sign.

Decoupler mode 3

Chemical shift region - aromatics ~150-110 ppm or integral data - aliphatics ~70-4 ppm

The samples were prepared by mixing the following components:

Jet fuel sample - 2 ml

NMR lock solvent - 1 ml deuterated chloroform

Chemical shift reference - 50 µl hexamethyldisiloxane

Relaxation agent - 50 mg 2.4 pentadione chromium

III derivative

Results

Sample Number	Carbon aromaticity, Car / Ctot
POSF-D-81-65	0.103
POSF-D-81-69	0.128

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^aThe gated proton decoupler was on during acquisition and off during delay.

SECTION 111

CONVENTIONAL FUELS

Jet turbine fuels are essentially hydrocarbon mixtures having inexact compositions that vary depending upon the kind and source of the crude oil used for their production. Despite these inherent compositional variations, different types of fuel can be consistently produced to meet a variety of exacting fuel specificiations. Conventional jet fuels include grades JP-4, JP-5, Jet-A, JP-7 and JP-8, with JP-4 currently being the most widely used fuel by the Air Force. The investigations described in this section were conducted to define the composition and properties of specific samples of these fuels for engineering applications, to support on-going studies or to aid in sclving operational problems related to the use of these fuels.

1. TRACE METAL TRANSFER FROM SUMP WATER TO FUELS

Sample Descriptions

Analyses were conducted in support of an Air Force program to determine the amount of dissolved metals transferred to jet fuels from tank water bottoms.

Nine fuel samples were analyzed for metals by atomic absorption (AA) and emission spectroscopy. Additionally, one of the fuels was dispersed with a synthetic sump (metal chloride) solution, and then was reanalyzed by AA after a period of 4 hours. The nine fuels and one fuel dispersion are described in Table 70.

Fuel numbers 1 through 6 and number 10 were semiquantitatively analyzed by the emission spectrographic technique for all metals present, then were quantitatively analyzed by AA for copper, lead, and cadmium. Samples 7. 8, and 9 were analyzed only for copper and cadmium, by AA.

TABLE 70. DESCRIPTION OF SAMPLES FOR TRACE METAL ANALYSES

MRC			·
Fuel No.	Cortainer Type	WPAFB	Sample Identification
1	Can	78-3346	Truck 73L-1180
2	Can	78-3348	Truck 73L-1147
3	Can	78 - 3348	Truck 72L-997
4	Can	78-3348	Truck 72L-1010
5	Can	F-16A-7	SN-780001
6	Can	F-16	SN, JP-4, fuel spec. 0748
7	Bottle	Standard (JRF plain (no metals added)
8	Bottle		d with 1.5 ppm copper and
9	Bottle	JP-4 spike	ed with 1.5 ppm copper and
16	Can/flask	after d	Truck 73L-1180 Fuel #1 ispersion with 87 g of a hloride solution

Sample Processing in Preparation for Analyses

SAND THE RESIDENCE OF STREET, ASSESSED ASSESSED.

Dispersion Technique for Fuel No. 10

A metal ion synthetic sump solution having the composition shown in Table 71 was dispersed with fuel number 10. The procedure for dispersal was simply a 15-second wrist oscillation of a pint bottle containing the fuel/solution mixture. The metal chloride solution and fuel phases were then allowed to separate overnight, after which the metal chloride solution was drained and discarded. The separated fuel was then acid extracted in preparation for the AA analyses.

Acid Extraction for AA Analyses

The fuels were extracted with high purity acid to sufficiently concentrate the metals in a nonvolatile form that could be quantified by AA. Two-milliliter quantities of metal-free concentrated

TABLE 71. CONCENTRATION OF SALTS AND METAL 10NS IN SYNTHETIC SUMP SOLUTION

Salt Formula	Salt, ppm	Metal Ion, ppm
CaCl;	50	18
CdCl ₂	1000	490
MgClaeHaO	50	6
NaCl	100	20
InCl ₂	10	4.7
CrCl3.6H2O	1	0.2
CuCla+2HaO	1	0.8
FeCla	5	1.7
MnClae4HaO	5	2.4
NiClao6HaO	1.	0.2
PbCla	1	0.7

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HCl^a were added to 250-g quantities of fuel number 10. The fuel/acid dispersions were shaken on a platform shaker for 10 min, after which 18-ml quantities of deionized and distilled water were added. The mixtures were shaken for an additional 20-min period and then the water and fuel phases were allowed to separate. The acidic water layers were analyzed for lead and/or copper and cadmium by AA.

Metals Concentration for Emission Spectrographic Analyses

Two-gram quantities of fuel were deposited into the carbon electrodes by the dropwise addition/fuel evaporation technique. This was followed by the addition of 10 mg lithium carbonate to simulate the matrix of the metal standards. The fuels were then analyzed against 10-mg quantities of lithium carbonate containing 10-, 100-, and 1,000-ppm quantities of about 50 different metals.

^aUltraRe hydrochloric acid; available from Hopkins and Williams; Essex, England.

The analyses of the 2-g quantities of fuel provided an effective concentration factor of 200. The estimated detection levels with this concentration factor were 0.05 ppm for copper, 1.0 ppm for cadmium, and 0.5 ppm for lead.

Results

The results of the emission and AA analyses for all the fuels are shown in Table 72. The emission analysis is estimated to have a precision of ±50%. A dash under a heading in the table indicates that none of the metal was detected. No copper, lead, or cadmium was detected in any of the samples by emission spectroscopy.

TABLE 72. EMISSION AND AA ANALYSIS RESULTS FOR FUELS

MRC	Brief									Acid	esults Extra	ets
Fuel	Descrip-		ssion A								uels,	
No.	tion	<u>Ng</u>	Si	<u>Fe</u>	<u> </u>	<u>Ca</u>	Na	<u>Ti</u>	<u>N1</u>	_ Cı	Pb_	Cd_
1	73L-1180	0.04	0.2	0.1	-	_	-	_	0-1	<0.016	0.071	0.014
2	73L-1147	0.4	7	0.1	0.2	3	0.1	_	_	<0.016	0.070	< 0.006
3	72L-997	1	20	0.1	0.4	2	2	0.1	-	<0.016	0.071	< 0.006
4	72L-101C	0.05	0.2	0.1	0.1	_	_	_	_	<0.016	0.045	<0.006
5	SN 780001	0.03	0.1	0.05	0.005	_		_	_	<0.016	0.053	0.018
6	SN, spec. 0748	0.04	1	0.2	0.2	-	-	-	-	<0.016	0.091	0.010
7	Plain JRF									<0.040		<0.015
8	Spiked JRF									1.93		0.74
9	Spiked JP-4									0.26		0.21
10	Conditioned No. 1	0.5	4	0.1	0.2	-	1	-	-	0.018	<0:04	1.06

The AA analysis is estimated to have a precision of ±2%. Values preceded by a "less than" (<) symbol indicate that none of this metal was detected at that level. The AA results showed that fuel number 8 contained copper and cadmium levels reasonably close to the expected 1.5-ppm levels, whereas fuel number 9 contained levels much lower. Fuel number 10 showed copper and cadmium levels that were increased over those in fuel number 1 in amounts proportional to the amounts in the metal chloride solution. Surprisingly, the amount of lead in fuel number 10 decreased to a nondetectable level.

2. TRACE METALS ANALYSIS OF JP-4 FUELS

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Five fuel specimens were analyzed for trace metals content, with particular attention being given to copper, lead, and cadmium. An initial emission spectrographic analysis was conducted to semi-quantitatively survey the metals content of each fuel. In order to accomplish this analysis, a 2-g portion of each fuel was deposited dropwise into the cup of a graphite electrode which was maintained at a temperature sufficient to vaporize the volatile hydrocarbons. Care was taken to insure that the electrode did not become hot enough to cause loss of volatile metals. The electrode was then used for analysis by the arc emission procedure using a Bausch and Lomb two-meter dual grating spectrograph.

Atomic absorption analyses were conducted on four fuels for copper and lead and one fuel for cadmium and lead, using an analytical method previously developed for that purpose (ref. 11). The procedure consists of extracting 150 to 200 grams of fuel with high purity hydrochloric acid (UltraR) followed by analysis of the combined acid and water rinses of the fuel. Aqueous standard solutions of metals were used for calibration. A reagent blank was analyzed along with each fuel. A Perkin-Elmer Model 303 atomic absorption spectrophotometer was used for the analysis.

Results of the emission spectrographic and atomic absorption analyses are presented in Tables 73 and 74.

3. PHYSICAL AND CHEMICAL PROPERTIES OF JP-5 FUEL

A JP-5 fuel was analyzed for certain physical and chemical properties by analytical techniques described in the appendix. The data were required for a NASA-generated computer program that predicts combustion parameters.

TABLE 73. SEMIQUANTITATIVE ANALYSIS OF FUELS FOR TRACE METALS BY EMISSION SPECTROSCOPY

			Co	ncentra	tion,	ppm	_	
Fuel	Mg	Si	Fe	Pb	Cd	Cu	Mn	Na
JP-4, A-4	0.005	0.05	0.005	<0.05	<0.1	<0.005	<0.01	<0.05
JP-4, A-7	0.03	0.05	0.01	<0.05	<0.1	<0.005	<0.01	<0.05
JP-4, Edwards								
Storage Tank 28	0.02	0.05	0.01	<0.05	<0.1	0.01	<0.01	<0.05
JP-4, Hercules			,					
88 + 88n (Edwards)	0.02	0.05	/0.02	<0.05	<0.2	0.01	<0.01	<0.05
JP-4, 1197	<0.05	<0.05	0.03	<0.05	<0.1	0.30	0.02	0.20

TABLE 74. ATOMIC ABSORPTION SPECTROPHOTOMETRIC METALS ANALYSIS

		Concentrat	ion, ppm
Fuel	Copper	Cadmium	Lead
JP-4, A-4	0.01	-	None detected
JP-4, A-7	0.007	-	0.030
JP-4, Edwards			
Storage Tank 28 JP-4, Hercules	0.005	-	0.026
88 + 88n (Edwards)	0.007	-	0.030
JP-4, 1197	-	<0.004	0.02

Note: Dash indicates analysis not conducted.

Vapor pressure and heat of combustion data are presented in Table 75. Gas chromatographic simulated distillation results are shown in Table 76, hydrocarbon type distribution values are in Table 77, and elemental analysis results from a commercial micro-analytical laboratory (Galbraith Laboratories, Inc.) are presented in Table 78.

4. EVALUATION OF JP-8 FROM SHALE OIL

Chemical property data were provided for the first batch of JP-8 fuel produced from shale oil crude at the Sohio Toledo refinery. The properties requested included elemental analysis, hydrocarbon-type distribution, simulated distillation by gas chromatography, and net heat of combustion.

TABLE 75. VAPOR PRESSURE AND HEAT OF COMBUSTION OF JP-5

Property	Determined value
Vapor pressure, mm Hg @ 100°F	31.0
Heat of combustion, Btu/lb	
Gross value, duplicate tests	19,752 19,784
Average	19,768
Net value	18,533

TABLE 76. GAS CHROMATOGRAPHIC SIMULATED DISTILLATION OF JP-5

Percent recovered	Boiling point °C	Boiling point
0.5, IBP	44	111
1	106	223
5	161	322
10	174	345
20	190	374
30	201	394
40	211	412
50	220	428
60	230	446
70	239	462
80	250	482
90	263	505
95	271	520
99	289	552
99.5, FBP	295	563

TABLE 77. HYDROCARBON-TYPE ANALYSIS OF JP-5 (MOD. ASTM D 2789)

Compound type	Volume, %
Paraffins	45.3
Monocycloparaffins	37.0
Dicycloparaffins	3.1
Alkylbenzenes	8.7
Indans and Tetralins	3.2
Naphthalenes	2.7

TABLE 78. ELEMENTAL ANALYSIS OF JP-5

	Weight percent	
	Carbon	Hydrogen
	82.80	12.92
	83.06	13.08
Mean	82.93	13.00

Elemental Analysis

The elemental analysis was conducted by a commercial microanalytical laboratory (Galbraith Laboratories, Knoxville). The results of duplicate analyses are shown below in weight percents in Table 79.

TABLE 79. ELEMENTAL ANALYSIS OF SHALE JP-8

Element	Duplicate	results, %	Average %
Carbon	86.22,	86.14	86.18
Hydrogen	13.76,	13.82	13.79
Oxygen	0.10,	0.14	0.12
Sulfur	0.086,	0.090	0.088
Nitrogen	0.0042,	0.0044	<u>0.0043</u> (43 ppm)
Total			100.22

Hydrocarbon Type Distribution

The hydrocarbon type distribution was determined by mass spectroscopy, using a procedure (ASTM D 2789-Modified) whereby characteristic mass fragments were summed to determine the various concentrations. The analysis was conducted on a CEC-21-103C mass spectrometer having a heated batch inlet. The distribution is shown in Table 80.

The total aromatic content of 20.5% is well within the specific maximum limit of 25% for JP-8. The indan/tetralin content for this fuel is somewhat higher than that found in most JP-8 fuels.

TABLE 80. HYDROCARBON TYPE ANALYSIS OF SHALE JP-8 (ASTM D 2789-Modified)

Hydrocarbon type	Volume %
Paraffin	46.7
Cycloparaffins	32.8
Dicycloparaffins	0.0
Alkylbenzenes	10.5
Indans/tetralins	9.2
Naphthalenes	0.8
Total	100.0

Simulated Distillation by Gas Chromatography

The boiling point distribution was determined by gas chromatograph raphy as described in ASTM D 2887. A gas chromatograph equipped with a flame ionization detector and a 6-foot by 1/8-inch OV-1 column was used for this work. The column was initially held 4 min at 50°C and then programmed up to 200°C at a rate of 8°C/min. A mixed hydrocarbon standard from C₄ to C₁₈ was used to correlate boiling point with retention time. Percent recovered values were determined from an integrating computer program which provided elapsed time, slice area, corrected area, accumulated area of the chromatogram, and percent recovered. The results are shown in Table 81.

Net Heat of Combustion

Net heat of combustion was determined by ASTM Method D 240, employing the oxygen bomb calorimeter. The 13.89% hydrogen value from the NMR analysis was used in the calculation rather than the very similar 13.79% value from the elemental analysis. The results are shown in Table 82.

TABLE 81. SIMULATED DISTILLATION OF SHALE JP-8 BY GC

Percent recovered	Retention time, min	Boiling °C	point °F
0.5	4.28	148	298
1	4.83	153.5	309
5	6.06	162.5	324.5
10	6.77	171	340
20	7.62	179	354
30	8.37	185.5	366
40	9.73	198	388
50	10.32	203.5	398
60	11.32	213	415
70	11.99	219.5	427
80	13.04	230	446
90	14.04	240	464
95	15.28	252	486
99	17.50	277	531
99.5	19.17	295.5	564

TABLE 82. HEAT OF COMBUSTION OF JP-5

Gross	Average	Net
19,811		
19,805		
	19,808	18,541

5. TRACE METALS ANALYSIS OF SHALE DERIVED JP-8

A series of five samples, which consisted of shale derived JP-8 stored in different containers under various conditions, were analyzed for trace metals content. The fuels were treated with high purity acid (metal-free) to extract trace metals according to methodology developed earlier for analysis of metals in fuels (ref. 11). An aliquot of the acidic extract was concentrated by dropwise addition and evaporation into the carbon electrodes used for emission spectrographic analysis.

Lower detection limits for metals of interest are as follows: Zn - 200 ppb; Pb - 20 ppb; Al - 4 ppb; Cu - 2 ppb; Sn - 10 ppb; Fe - 6 ppb. Analytical results are shown in Table 83 below.

TABLE 83. EMISSION SPECTROGRAPHIC ANALYSIS OF FUELS

		Parts-per-	billion
Fuel sample		Copper	Tin
JP-8, JP-8, JP-8,	fuel rinsed, lined can fuel rinsed, unlined can solvent rinsed, lined can solvent rinsed, unlined can control	- 2 4 6	14 - -

Dash (~) indicates none detected. No Zn, Pb, Al or Fe was detected.

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6. CHEMICAL AND PHYSICAL PROPERTIES OF AMRL JP-8 FUEL

Chemical and physical property determinations were performed on a JP-8 fuel sample to provide data required by the Air Force Civil and Environmental Engineering Office at Tyndall Air Force Base, Florida.

Kinematic viscosity, density, vapor pressure and surface tension were determined as a function of temperature. Net heat of combustion was measured by oxygen bomb calorimetry. Simulated distillation was conducted by gas chromatography, and mass spectrometry by ASTM Method D 2425 was used for hydrocarbon type distribution. All test methods a described in the Appendix. The JP-8 fuel was fractionated before the hydrocarbon type analysis, and this is described below.

Table 84 lists data or density, kinematic viscosity, surface tension, and vapor pressure; Table 85 presents heat of combustion values; and Table 86 provides simulated distillation data. Figure 72 shows the viscosity/temperature relationship.

TABLE 84. PHYSICAL PROPERTIES OF AMRL JP-8
Temperature.

Property	°F	
Density, g/cc	32 70 100	0.8172 0.8018 0.7894
Kinematic viscosity, centistokes	0 77 100	4.553 1.737 1.417
Surface tension, dynes/.m	32 70 100	28.15 26.18 24.58
Vapor pressure, mm Hg	32 70 100	3.3 11.0 25.7

TABLE 85. HEAT OF COMBUSTION OF AMRL JP-8

Gross,	Btu/lb	Net, Btu/lb
Duplicate	Average	Average

19,657 19,710

19,684

18,407

TABLE 86. GAS CHROMATOGRAPHIC SIMULATED DISTILLATION OF AMRL JP-8

Percent	Temper	cature
Recovered	°C	°F
0.5 (IBP)	102	216
1	115	239
5	144	291
10	165 182	329 360
30	194 202	381 396
40 50	210	410
60	218	424
70	227	441
80	238	460
90	256	493
95	270	518
99	294	561
99.5 (FBP)	303	577

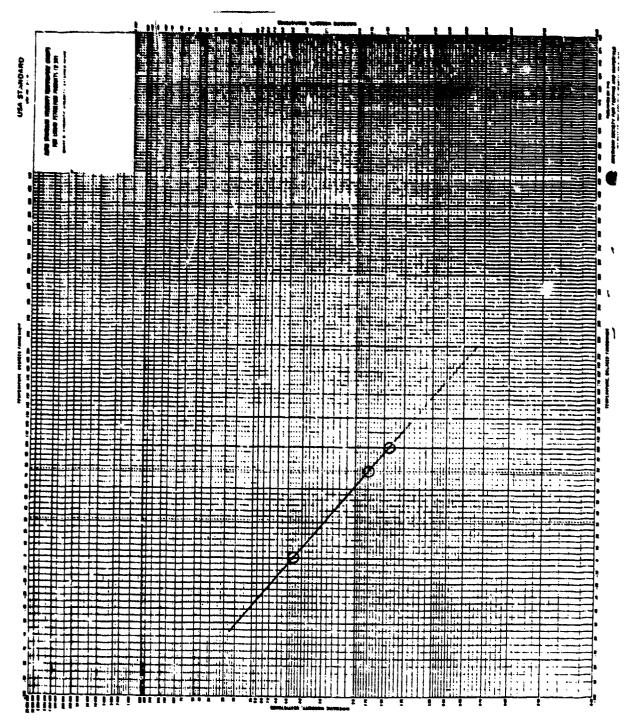


Figure 72. Viscosity/temperature plot for AMRL JP-8.

A hydrocarbon type analysis was conducted on the fuel using the approach of isolating aromatic and paraffinic fractions by elution chromatography (ASTM D 2549) and then analyzing each fraction by a mass spectrometric procedure (ASTM D 2425). The preliminary separation was conducted on a 50-cm column of activated bauxite and silica gel by sequential elution with pentane, ethyl ether, chloroform, and ethyl alcohol.

The mass spectrometric analysis provided quantitative information on 11 hydrocarbon types. The analysis was based on the summation of characteristic fragment mass intensities to determine the concentration of various hydrocarbon types.

By summation of various combinations of more than 110 mass spectral peaks, matrices consisting of sets of 5 linear simultaneous equations for the saturate fraction and 10 for the aromatic fraction were constructed. Alkylbenzenes, which were calculated in the saturate fraction and paraffins calculated in the aromatic fraction, demonstrated the completeness of the chromatographic separation. Computer solution of each matrix yielded weight percent of each compound type. Hydrocarbon type data are presented in Table 87.

TABLE 87. HYDROCARBON-TYPE DISTRIBUTION OF AMRL JP-8 BY ASTM D 2425

Compound type	Weight percent
Paraffins	42.29
Cycloparaffins	35.28
Dicycloparaffins	9.10
Tricycloparaffins	1.64
Alkylbenzenes	8.57
Indans and tetralins	1.83
Indenes	0.04
Naphthalene	0.34
Naphthalenes	0.90
Acenaphthenes	_a
Acenaphthylenes	•
Tricyclic aromatics	0.01

^aDash indicates none was detected.

7. DIELECTRIC CONSTANT OF JP-8 FUEL WITH ANTISTATIC ADDITIVES

The dielectric constant as a function of temperature was determined for JP-8 with and without antistatic additives. The purpose of the work was to determine whether the presence of these additives in the fuel has a significant effect on its dielectric constant.

A General Radio 1615A capacitance bridge and guard circuit were used for the measurements. The bridge signal generator was a General Radio 1311A audio oscillator and the bridge detector was a Type 1232A tuned amplifier and null detector.

Capacitance measurements were made in a three-terminal guarded cell relative to air at the same temperature.

Dielectric constant values are given in Table 88 below.

TABLE 88. DIELECTRIC CONSTANT OF JP-8 WITH/ WITHOUT ANTISTATIC ADDITIVES

	Tem	perature,	°F_
	0	74	100
JP-8, Tank F-3	2.192	2.144	2.127
JP-8 + 2PPM ASA-3	2.186	2.137	2.120
JP-8 + 2PPM S-450	2.181	2.132	2.115

8. CHEMICAL PROPERTIES OF ENGLISH JP-8 FUEL

Hydrocarbon type and distillation temperature range data were provided for a sample of JP-8 from Bendix fuel control division. The effect of this fuel on elastomeric seals was evaluated as part of another Air Force program.

Simulated distillation was conducted according to ASTM procedure D 2887 to determine boiling point range. A gas chromatograph equipped with a 3% OV-1 column and a hydrogen flame ionization detector was used for this work. Hydrocarbon type analysis by mass spectrometry was conducted according to ASTM Method D 2789-71.

Data are presented in Tables 89 and 90.

TABLE 89. SIMULATED DISTILLATION OF ENGLISH JP-8 BY GAS CHROMATOGRAPHY

Percent recovered	Englis BP, C	h JP-8 BP, °F
0.5, IBP	92	198
1	104	219
5	141	206
10	156	313
20	170	338
30	180	356
40	190	374
50	198	388
60	209	408
70	218	424
80	231	448
90	245	473
95	254	489
99	270	518
99.5, FBP	276	529

TABLE 90. HYDROCARBON TYPE DISTRIBUTION OF ENGLISH JP-8 BY MASS SPECTROMETRY

Compound type	Volume, 3
Paraffins	55.4
Monocycloparaffins	30 . 1
Dicycloparaffins	-
Alkylbenzenes	11.7
Indans and tetralins	2.0
Naphthalenes	0.8

9. ANALYSIS OF TWO BROAD SPECTRUM ERBS FUELS

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Two samples of experimental reference broad spectrum fuels were analyzed in support of Alternate Source Development.

Simulated distillation was conducted by gas chromatography as described in ASTM D 2887. A gas chromatograph, equipped with a flame ionization detector and a column of SP-2100, was used for this work. Data are presented in Table 91.

TABLE 91. SIMULATED DISTILLATION OF BROAD SPECTRUM FUELS

	ERB	S #1	ERB:	5 #2
	Boiling	Point	Boiling	Point
Percent Recovered	<u>•c</u>	•F	°C	°F
0.5, IBP	132	270	129	264
1	. 141	286	140	284
5	163	325	162	324
10	173	343	173	343
20	188	370	188	37 0
30	197	387	197	387
40	208	466	208	406
50	218	424	218	424
60	230	446	231	448
70	240	464	244	471
80	258	496	261	502
90	287	549	290	554
95	313	595	315	601
99	355	671	366	691
99.5, FBP	365	689	377	711

Hydrocarbon type analyses were conducted using ASTM procedure D 2789-71. Data are presented in Table 92.

Nitrogen analyses were obtained from Galbraith Laboratories, Inc. (Knoxville, TN). Data are presented in Table 93.

TABLE 92. HYDROCARBON TYPE DISTRIBUTION OF BROAD SPECTRUM FUELS BY MASS SPECTROMETRY

		percent
Compound type	ERBS #1	ERBS #2
Paraffins	46.6	46.8
Monocycloparaffins	32.6	32.4
Dicycloparaffins	-	-
Alkylbenzenes	9.5	9.5
Indans and Tetralins	5.3	5.3
Naphthalenes	6.0	6.0

TABLE 93. ELEMENTAL ANALYSIS OF BROAD SPECTRUM FUELS

	Nitroge	n, ppm
	ERBS #1	ERBS #2
	84	85
	82	78
Mean	83	. 82

10. HYDROCARBON-TYPE DISTRIBUTION IN MASA JET "A" AND DIESEL FUELS

Chemical property data were required for two fuel specimens being studied independently at AFAPL and NASA Lewis Research Center. A hydrocarbon-type distribution analysis was requested along with an independent check for total naphthalenes content by conventional ASTM procedure D 1840.

Analytical Procedures

Modified ASTM Method D 2789 was used to determine the concentration of six different hydrocarbon types present in two fuels. Mass spectra of the fuels were recorded, and summations of characteristic mass fragments for the hydrocarbon types were related to concentrations by means of calibration mixtures. ASTM Method D 1840 was used to determine the total concentration of naphthalene, acenaphthene, and alkylated derivatives of both of these compounds. Ultraviolet spectrophotometry is employed in this method, with absorbance at 285 nm being measured for known concentrations of fuel in cyclohexane. Phenanthrenes, dibenzothiophenes, biphenyls, benzathiophenes, and anthracenes interfere with the method if present and add to the apparent naphthalene content.

Results

The analytical results from both procedures are shown in Table 94.

TABLE 94. RESULTS OF HYDROCARBON TYPE AND NAPHTHALENES ANALYSES FOR TWO FUELS

		Concer	ntrations	of compou	ind types
•		NASA	Jet "A"	NASA ć	liesel fuel
Compound types	Method	(Vcl %)	(Wt %)	(Vol %)	(Wt %)
Paraffins	MS ^a	45.8		38.4	
Monocyclo-paraffin	MS	37.4		27.0	
Dicyclo-paraffin	MS	1.0		0.8	
Alkylbenzenes	MS	9.0		8.4	
Indans and Tetralins	MS	4.4		10.5	
Naphthalenes	MS	2.4	3.1	14.9	19.2
Naphthalenes	UV Absorption		2.4, 2.4 ^b		25.6, 25.8 ^b

^aMass Spectrometric.

The mass spectral hydrocarbon type analysis yields results in liquid volume percent, while the naphthalene determination (ASTM D 1840) is computed in weight percent. In order to compare data, the volumetric naphthalene value was converted to weight percent. The higher naphthalene content for the diesel fuel by ASTM D 1840 indicates the probable presence of interfering compounds.

^bDuplicate analyses.

11. METALS ANALYSIS OF SHALE-DERIVED JET FUEL

Shale-derived jet fuel, which was shipped in an unlined metal can, was found during evaluation at AFAPL to fail the thermal stability tests. A metals analysis was requested to determine if contamination from the sample container was responsible for the fuel instability.

An emission spectrographic analysis of the fuel sample, labeled 9105-1, showed the presence of 200 ppb tin and 8 ppb nickel. No other metals were detected. The tin source may be the solder seam in the fuel can, and its presence is the likely cause of the poor thermal stability.

12. VAPOR PRESSURE OF JP-4 FROM BOTH PETROLEUM AND SHALE OILS

Vapor pressure was determined for petroleum- and shale-derived JP-4 samples by use of the microvapor pressure apparatus and procedure described in ASTM Method D 2251 and reference 9. Results are presented below.

	Vapor press	sure, mm Hg
Sample number	135°F	150°F
145-400-792033	229	297
HRI-LO-2054	150	191

13. GC, MS, AND NMR ANALYSES OF EIGHT SHALE-DERIVED JET FUELS

Samples of eight shale-derived jet fuels were analyzed for boiling range distribution by GC-simulated distillations, hydrocarbon-type distribution by mass spectrometry, and aromaticity by carbon-13 and proton high-resolution NMR spectroscopy. The eight shale-derived jet fuel samples were identified as follows.

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1. HRI-LO-2054 (80-PHJ-084A)

5. UOC-9H15-6

2. HRI-LO-2057 (80-PHJ-084B)

6. UOC-9105-1

3. UOC-A-1655 Rehydrotreated

7. UOC-9H15-7

4. UOC-B-1656 Single Pass

8. UCC-9105-2

The simulated distillations were conducted by gas chromatography as described in ASTM Method D 2887. A gas chromatograph equipped with a flame ionization detector and a column of 3% OV-1 on Chromosorb W was used for this work. Data are presented in Table 95.

The hydrocarbon-type analyses were conducted using the modified ASTM procedure D 2789 for all samples. Additionally, Monsanto Method 21-PQ-38-63 was used for two samples. Data are presented in Table 96.

The carbon-13 NMR analyses were conducted to determine carbon aromaticity, which is a ratio of the aromatic carbon to the total carbon in the fuel. Likewise, the proton NMR analyses were conducted to determine hydrogen aromaticity, which is a ratio of the aromatic hydrogen in the fuel to the total hydrogen. Using these aromaticity values and the ratio of total hydrogen to total carbon in the fuels (obtained from elemental hydrogen analyses conducted at AFAPL), the ratio of aromatic hydrogen to aromatic carbon was then calculated for the eight fuels. This ratio was to be used to correlate the composition of the fuel with its combustion behavior. The total area under the peaks in specified spectral regions of the proton NMR curves was also provided for use in a computer program.

NMR Analytical Parameters

The NMP analyses were conducted on a Varian CFT-20 Fourier transform spectrometer containing a Varian 602L computer for data acquisition, data reduction, and system control. The spectrometer

TABLE 95. SIMULATED DISTILLATION BY GAS CHROMATOGRAPHY

	UOC-9105-2	94	293	307	345	383	424	460	9	5.31	568	294	626	662	693	747	
	6-00n	(۲	145	153	174	195	218	238	258	277	588 588	312	330	350	367	397	
	UOC-9H15-7	.	289	304	347	382	442	9	511	543	572	595	621	2	699	718	,
	5-20D	ပ	143	151	175	196	228	249	366	284	8	313	327	343	354	381	900
	UOC-9105-1	-	124	136	205	241	282	307	333	347	365	395	397	419	428	457	471
	6-0 00 00 00	•	25	8	96	116	139	153	167	175	185	196	. 203	215	220	236	244
	ос-9H15-6	<u>a.</u>	180	192	241	262	293	318	334	347	361	381	392	414	423	446	464
	5-200	မ	83	68	116	128	145	159	168	175	183	194	200	212	217	230	240
	JOC-B-1656	2-1	135	151	208	243	275	304	331	351	374	394	421	442	457	486	502
	-TOC	ပ	57	99	86	117	135	151	166	177	190	201	216	228	236	252	261
	DOC-A~1655	a.	131	149	212	250	291	322	345	367	38,	406	423	442	455	484	495
		ပ	\$ 2	9	100	121	144	191	174	186	196	208	217	228	235	251	257
	HR10-2057 80-PHJ -084B)		83	97	203	234	262	300	331	356	383	405	432	464	496	529	579
	(80-PHJ	ပ	28	36	95	112	128	149	166	180	195	207	222	240	258	293	304
7300	-084A)	p-	11	93	190	225	277	309	340	363	387	403	426	457	491	577	585
1300 VI 100	(80-PE)	30	25	34	88	107	136	154	171	134	197	506	219	336	255	303	307
	Percent	Becovered	0.5 (IBP)	1	in.	Ju	20	30	4 0	20	9	70	80	06	95	66	99.5 (FBP)

TABLE 96. HYDROCARBON-TYPE ANALYSIS

ACTOR AND THE PROPERTY OF THE

	74				Volume	Volume Percent				
	HRZ-LO-2054 (80-PHJ-064A)	MEI-LO-2057 [®] (80-797-0848)	UOC-A-1655		noc-a-1656 ⁶ uoc-9415-6	uoc-9105-1	10C-9415-7 Method 1 ^a	UC-9415-7 Method 2b	UDC-9415-7 UDC-9105-2 UDC-9105-2	UOC-9105-2 Method 2
Paraf fins	46.5	4.8	46.0	50.7	46.7	49.5	46.0	46.2	45.4	45.5
Monocycloparaffins	42.5	42.9	50.3	46.8	42.6	39.5	37.2	36.36	37.4	30.1°
Dicycloparaffins	1.9	1.1	•	۰	•	•••	2.1		1.4	
Alkylbenzene	6.2	9.1	3.0	2.0	9.0	7.	0.9	6.9	6.8	7.6
Indans/Tetralin	2.9	2.1	0.0	v. 0	1.7	2.2	7.4	7.0	0.0	1.7
haphthalenes	Trace	Trace	•	•	•	•	1.3	6.0	1.3	1.1

Prathod 1. Modified ASTM D2789-71

bethod 2. Wonsanto 21-PQ-38-63.

This method combines mon. - and dicycloparaffins into a single value.

 $(\frac{1}{2},\frac{1}{2})$

was operated at 20 megahertz for the ¹³C analyses and 79.54 megahertz for the proton NMR analyses. The following instrument conditions were utilized for the two analyses:

Type NMR Analysi	.s	Carbon-13	Proton (1H)
Sample Probe		10 mm	5 mm
Sweep Width		4,000 Hz	1,000 Hz
Number of Transients		1,000	50
Acquisition Time		1.023 s	4.095 s
Pulse Width 90°		17 µs	24 µs
Pulse Delay		5 ຮ	8 s
Homo-Spoil Time ^a		Not on	8 ms
Data Points		8,192	8,192
Decoupler Mode		3 ^b	***
Chemical Shift Regions	Aromatics	~150-110 ppm	8.3-6.5 ppm
for Integral Data	Aliphatics	~7C-4 ppm	4.0-0.2 ppm

^aHomospoil was on during pulse delay.

The analytical samples were prepared for carbon-13 analysis by mixing the following components:

Jet Fuel Sample - 1 ml

NMR lock solvent - 0.5 ml deuterated chloroform ("100%" CDCl₃) Chemical shift reference - 0.5 ml hexamethyl disiloxane Relaxation agent - 0.5 mg 0.4-pentanedione chromium III derivative

For the proton NMR analysis, a 20 μ l quantity of the above mixture was added to 0.5 ml additional CDCl₃.

b
The gated proton decoupler was on during acquisition and off during delay.

Calculations and Results

The carbon aromaticity values were obtained by integrating the peak areas in the aromatic region of the ¹³C spectra relative to the total peak area in the spectra. The hydrogen aromaticities were obtained in the same manner from peak areas in the proton NMR spectra. The ratio of aromatic hydrogen to aromatic carbon was obtained from the following equation:

$$\frac{H_{ar}}{C_{ar}} = \frac{C_{T}}{C_{ar}} \times \frac{H_{ar}}{H_{T}} \times \frac{H_{T}}{C_{T}}$$
I II III

where I = the inverse of carbon aromaticity determined by 13 C NMR

II = the hydrogen aromaticity determined by proton NMR

III = total hydrogen/carbon ratio determined from percent
 hydrogen data provided by AFAPL

The aromaticity values for the eight fuels and their aromatic and total hydrogen/carbon ratios are presented in Table 97. The integrated areas for the specified spectral regions of the proton NMR spectra are listed in Table 98.

In general, no specific compounds were identified by the ¹³C and proton NMR analyses. However, the one exception was fuel HRI-LO-2057 which was observed to contain a significant quantity of toluene.

<u>Comparison of Carbon Aromaticities With Values From Mass</u> Spectrometric Analyses

Carbon aromaticity was calculated from the hydrocarbon-type analysis data. The volume percents were recomputed to weight percents and an average compound structure was formulated for each fuel constituent using an average carbon number determined from the GC

TABLE 97. AROMATICITY VALUES AND HYDROGEN/CARBON RATIOS

Sample designation	Carbon aromaticity, Car /CT	Hydrogen aromaticity, Har/HT	Percent H _T b	$\frac{\text{Mole ratio}_{\text{C}}}{\text{H}_{\text{T}}/\text{C}_{\text{T}}}$	Mole ratio H _{ar} /C _{ar}
HRI-LO-2054	0.062	0.022	14.43	2.01	0.714
HRI-LO-2057	0.099	0.039	14.22	1.98	0.774
UOC-A-1655	0.045	0.018	14.68	2.05	0.821
UOC-B-1656	0.033	0.009	14.82	2.07	0.560
UOC-9H15-6	0.086	0.029	14.34	1.99	0.667
UOC-9105-1	0.086	0.031	14.36	2.00	0.726
UOC-9H15-7	0.086	0.033	13.77	1.90	0.726
UOC-9105-2	0.089	0.040	13.70	1.89	0.845

These values may not be as accurate as the hydrogen aromaticity values because of a low signal/noise ratio in the aromatic region of the 13C spectrum.

TABLE 98. INTEGRATED AREAS OF SPECIFIED PROTON NMR SPECTRAL REGIONS

				Spect	ral Region	ns_		
	HMONO	HDI	HTRI	HALP 1	HALP 2	H BETA	H GAMA	H BETH
	6.6-	7.3~	7.8-	2.3-	1.9-	1.9-	1.0-	1.90-
	7.3,	7.8,	8.3,	4.0,	2.3,	1.0,	0.5,	1.65,
Sample	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
HRI-LO-2054	6	$\mathtt{ND}^{\mathbf{b}}$	ND	2	7	139	111	9
HRI-LO-2057	8	ND	ND	1	7	105	84	6
UOC-A-1655	3	1	ND	ND	4	121	98	7
UOC-B-1656	2	ND	ND	1	2	125	96	6
UOC-9H15-6	6	ND	ND	1	8	108	82	5
UOC-9105-1	6	ND	ND	ИD	6	99	83	5
UOC-9H15-7	6	1	ND	3	6	117	74	6
UOC-9105-2	7	1	1	7	8	128	74	8

The listed integral areas are accurate to ± 0.5 . Fractional values are rounded to the nearest whole number by the data system.

bData from AFAPL by ASTM D 3701.

 $^{^{} extsf{C}}$ Total carbon data were obtained by subtracting the % $^{ extsf{H}}_{ extsf{T}}$ from 100.

bND shows a total area less than 0.5.

simulated distillation analysis. The percents of aromatic and aliphatic carbon in each fuel constituent were calculated from the weight percentages and compound structures. The carbon aromaticity of the entire fuel was then calculated from the ratio of total aromatic carbon to total carbon in the fuel. These calculated carbon aromaticity values and all data used in the calculations are shown in Table 99. The calculated carbon aromaticity values and those obtained from ¹³C NMR are compared in Table 100.

Conclusions

In general, the aromaticity values calculated from the hydrocarbon type analyses were slightly lower than the corresponding values calculated from the ¹³C NMR analyses. However, the overall agreement of data was good considering the diversity of the two methods and the experimental error inherent in the component parts of each analysis. The relative differences in the fuels were highlighted by the aromaticity values obtained by both analytical approaches.

It is interesting to note the generally good correlation of the total hydrogen values with aromaticity values. As expected, the hydrogen content increased as aromaticity decreased. The only notable exception occurs for fuel number HRI-LO-2057. This fuel showed the highest aromaticity of all the fuels by all three analytical techniques, yet it did not have the lowest hydrogen content. Coincidentally, this was also the only fuel that was shown earlier to contain toluene.

The H_{ar}/C_{ar} value, given in the last column of Table 97, is useful in evaluating the nature of a fuel's aromatic fraction. However, both C_{ar}/C_{T} and H_{ar}/C_{ar} values should be used for correlation with the fuels combustion characteristics.

CARBON AROMATICITY FROM HYDROCARBON TYPE ANALYSES TABLE 99.

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Carbon aromaticity			0.072	0.089	0.031
Mole percent aliphatic carbon		43.6 2.0 2.4 1.2	92.8	41.9 43.7 1.2 3.5 0.8 91.1	43.6 51.8 0.0 1.2 0.3
Mole percent aromatic carbon		0 0 7 7 8 7 8 7	7.2	0 0 7.1 1.8 8.9	0 0 0 0.7 3.1
Average compound		C10H22 C10H20 C10H18 C9H12		C ₁₀ H ₂₂ C ₁₀ H ₂₀ C ₁₀ H ₁₈ C ₉ H ₁₂ C ₉ H ₁₀	C10H22 C10H20 C10H18 C9H12 C9H10
Weight percent	Fuel HRI-LO-2054	44.2 43.6 1.9 6.9 4.5	Fuel HRI-LO-2057	42.5 43.8 1.2 10.1 2.4 -A-1655	44.1 51.7 0.0 3.3 0.9
Volume percenta	Fuel HRI	46.5 42.5 1.9 6.2 2.9	Fuel HRI	44.8 42.5 42.9 43.8 1.1 1.2 9.1 10.1 2.1 2.4 Fuel UOC-A-1655	46.0 50.2 0.0 3.0 8.0
Fuel constituents		Paraffins Monocycloparaffins Dicycloparaffins Alkylbenzenes Indans/Tetralin	Total	Paraffins Monocycloparaffins Dicycloparaffins Alkylbenzenes Indans/Tetralin Total	Paraffins Monocycloparaffins Dicycloparaffins Alkylbenzenes Indans/Tetralin Total

#eight	
# Mole percent percent percent percent percent percent carbon da.3 CloH22	
C10H22 0 48.3 C10H20 0 0.0 C10H18 0 0.0 C9H10 0 0.2 C9H10 0.5 C10H2 0 0.5 C10H20 0 43.7 C10H20 0 0.0 C9H12 1.4 0.7 C10H20 0 0.0 C10H20 0 0.0 C10H20 0 0.0 C10H20 0 0.0 C10H20 0 0.5 C10H20 0 0.5 C10H20 0 0.5 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 1.9	Volume Dercent
C10H22 0 48.3 C10H20 0 48.6 C10H18 0 0.0 C9H12 1.6 0.2 C10H20 0 0.5 C10H20 0 43.7 C10H18 0 0.0 C9H12 1.4 0.7 C10H20 0 43.7 C10H20 0 43.7 C10H20 0 0.0 C10H20 0 0.0 C10H20 0 0.0 C10H20 0 0.5 C10H20 0 0.5 C10H20 0 0.5 C10H20 0 0.5 C10H20 0 0.5 C10H18 6.5 3.3	1 9
C10H22 0 48.3 C10H20 0 48.6 C10H18 0 0.0 C9H10 0.5 0.2 C9H10 0.5 0.2 C10H20 0 43.7 C10H20 0 43.7 C10H12 0 0.0 C9H10 1.4 0.7 C10H22 0 46.4 C10H22 0 46.4 C10H22 0 46.5 C10H18 0 0.5 C10H18 0 0.5	Fuel Uoc-B-
C10H20 0 48.6 C10H18 0 0.0 C9H12 1.6 0.2 C9H10 0.5 0.2 C10H22 0.5 0.2 C10H22 0 43.7 C10H18 0 0.0 C9H10 1.4 0.7 C10H22 0 46.4 C10H22 0 46.4 C10H18 0 0.5 C9H10 0 0.5 C9H10 0 0.5 C9H10 0 0.5 C9H10 0 0.5 C9H10 0 0.5 C9H10 0 0.5 C9H10 0 0.5	
C10H18 0 0.0 C9H12 1.6 0.2 C9H12 1.6 0.2 C10H20 0 43.7 C10H20 0 43.7 C10H18 0 0.0 C10H18 0 0.0 C10H22 0 46.4 C10H20 0 46.4 C10H20 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 6.5	4
CoH12 1.6 0.2 CoH10 0.5 0.2 CloH22 0 43.7 CloH20 0 43.7 CloH18 0 0.0 CloH22 0 43.7 CoH12 1.4 0.7 CloH22 0 46.4 CloH22 0 46.4 CloH22 0 46.4 CloH22 0 6.5 CloH18 0 0.5 CoH12 0 6.5 CoH12 0 6.5	
C10H22 0 43.7 C10H20 0 43.7 C10H20 0 0.0 C9H12 7.0 3.5 C9H12 1.4 0.7 C10H22 0 46.4 C10H20 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 6.5	2.0
C10H22 0 43.7 C10H20 0 43.7 C10H18 0 0.0 C9H12 7.0 3.5 C9H10 1.4 0.7 8.4 91.6 C10H20 0 46.4 C10H20 0 46.5 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 6.5	
C10H22 0 43.7 C10H20 0 43.7 C10H18 0 0.0 C9H12 7.0 3.5 C9H10 1.4 0.7 8.4 91.6 C10H20 0 46.4 C10H20 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 6.5	Fuel UOC-9H15-6
C10H20 0 43.7 C10H18 0 0.0 C9H12 7.0 3.5 C9H12 1.4 0.7 8.4 91.6 C10H20 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H12 6.5	46.7 44
C10H18 0 0.0 C9H12 7.0 3.5 C9H10 1.4 0.7 8.4 91.6 C10H20 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H1C 1.9 0.9	•
CoH12 7.0 3.5 CoH10 1.4 0.7 8.4 91.6 C10H22 0 46.4 C10H18 0 0.5 C9H12 6.5 3.3 C9H10 1.9 0.9	
C ₁₀ H ₂₂ 0 46.4 C ₁₀ H ₂₀ 0 46.5 C ₁₀ H ₁₈ 0 6.5 C ₉ H ₁₂ 6.5 3.3 C ₉ H ₁₀ 1.9 0.9	9.0 10 1.7 2
C ₁₀ H ₂₂ 0 46.4 C ₁₀ H ₂₀ 0 40.5 C ₁₀ H ₁₈ 0 0.5 C ₉ H ₁₂ 6.5 3.3 C ₉ H ₁₀ 1.9 0.9	
C ₁₀ H ₂₂ 0 46.4 C ₁₀ H ₂₀ 0 40.5 C ₁₀ H ₁₈ 0 0.5 C ₉ H ₁₂ 6.5 3.3 C ₉ H ₁₀ 1.9 0.9	Fuel UOC-9105-1
C ₁₀ H ₂₀ 0 40.5 C ₁₀ H ₁₈ 0 0.5 C ₉ H ₁₂ 6.5 3.3 C ₉ H ₁₀ 1.9 0.9	
C ₁₀ H ₁₈ 0 0.5 C ₉ H ₁₂ 6.5 3.3 C ₉ H ₁₀ 1.9 0.9	
C9H ₁₂ 6.5 3.3 C9H ₁₀ 1.9 0.9	
7.10 Time	4. °C
	1

(continued)

TABLE 99 (continued)

				Mole	Hole	
Fuel Constituents	Volume percent	Weight, percent	Average compound	aromatic carbon	aliphatic carbon	Carbon aromaticity
	Alfel IIO	Fire 1 110C-9H1 5-7				
		7 7 7 7				
Paraffins	46.0	43.3	C1 EH22	0	42.8	
Monocycloparaffins	37.2	37.7	C ₁₅ H ₃₀	0	37.6	
Dicycloparaffins	2.1	2.1	C1sH2g	0	2.1	
Alkylbenzenes	6.0	9.9	C14H22	2.9	3.8	
Indans/Tetralin	7.4	8.6	C14H20	3.9	5.1	
Naphthalenes	1.3	1.7	C14H16	0.8	1.0	
Total				7.6	92.4	0.076
	Fuel UO	Fuel UOC-9105-2				
Paraffins	45.4	42.5	C1 EH32	0	42.0	
Monocycloparaffins	37.4	37.8	C16H30	0	37.6	
Dicycloparaffins	1.4	1.4	C ₁ sH ₂ s	0	1.4	
Alkylbenzenes	6.5	7.2	C14H22	3.2	4.2	
Indans/Tetralin	8.0	9.4	C14H20	4.2	5.6	
Naphthalenes	1.3	1.7	C14H16	0.8	1.0	
Total				8.2	91.8	0.082

Results from mass spectrometric hydrocarbon type analyses.

b Calculated using density values.

CDetermined using an average carbon number obtained from simulated distillation analyses.

dcalculated from structure of average compound.

Calculated by dividing the percent aromatic carbon by the percent total carbon in the fuel.

TABLE 100. COMPARISON OF CARBON AROMATICITY VALUES

Fuel	Values from 13C NMR	Values from mass spectrometry
HRI-LO-2054	0.062	0.072
HRI-LO-2057	0.099	0.089
UOC-A-1655	0.045	0.031
UOC-B-1656	0.033	0.021
UOC-9H15-6	0.086	0.084
UGC-9105-1	0.086	0.084
UOC-9H15-7	0.086	0.076
UOC-9105-2	0.089	0.082

^aUsing average carbon numbers from simulated distillations by GC.

14. VAPOR COMPOSITION OF JP-4 AND JP-8 FUELS IN EQUILIBRIUM WITH THEIR BULK LIQUIDS

Fuel flammability studies conducted within the Fire Protection Branch of the Aero Propulsion Laboratory generated a need for information concerning the precise composition of vapors in equilibrium with bulk aircraft fuels. Therefore, a study was conducted to determine equilibrium vapor compositions of two fuels, a JP-4 (Tank 15) and a JP-8 (Tank F-3), at various temperatures. The procedures and results of this work are documented below.

Procedure

A vapor equilibration system was constructed which consisted essentially of a 400-ml glass vessel immersed into a constant temperature bath. A quantity of 200 ml of fuel was placed into

the vessel, giving a 1:1 ratio in the volumes of fuel and vapor. A 1-ml sampling loop was attached to the system for withdrawing vapor samples. The loop could be evacuated by means of a vacuum pump prior to sampling the vapor. The head space and vapor sample volumes were selected in order to minimize disruption of the equilibrium by removal of vapor phase.

Vapor analyses were conducted using a Hewlett-Packard GC/MS unit with attached data system, and a Perkin Elmer 3920 gas chromatograph having a 50-meter glass capillary column coated with SF-96 liquid phase. The mass spectral data were recorded in the continuous scanning mode and were used primarily for compound identification. The high-resolution GC system was used for quantitation by means of a hydrogen flame ionization detector (FID). In some cases, a precise structural configuration could not be unambiguously determined from the mass spectrum alone. Reference hydrocarbons were used to establish a boiling point/retention time relationship which was then used to aid in selecting the correct compound from the several possible configurations.

Results

Vapor compositions are presented in Tables 101 and 102 at four different temperatures. Linear and cyclic paraffins, which comprised the bulk of the vapor phase, responded uniformly by FID on essentially a carbon number basis making the response proportional to weight composition. These compounds were consequently assigned a weight response factor of unity. Several aromatic constituents were found in the vapor phase and individual response factors for these compounds were applied in calculating compositions.

All components present at concentrations of 1% or greater in the vapors were reported. These components accounted for a major part of the vapor. The remaining portion consisted of low levels of a myriad of compounds. Each component of the fuel, even though it

TABLE 101. VAPOR COMPOSITIONS OF JP-4, TANK 15

Component		Weight Percent			
Component	0°£	35 ° F	70°F	105°F	
Cyclopropane	5.1	4.4	3.3		
2-Methylpropane	8.0	6.4	5.7	1.5	
Butane	15.6	11.9	12.0	3.5	
2-Methylbutane	16.6	13.3	15.4	4.3	
Pentane	14.6	10.8	13.4	4.2	
2,3-Dimethylbutane	4.8	5.0	5.8	1.3	
3-Methylpentane	3.0	3.3	4.1	1.5	
Hexane	7.5	5.9	7.4	3.9	
2,2-Dimethylpentane	2.3	2.2	2.4	1.4	
Cyclohexane	2.3	2.3	2.6	1.5	
3,3-Dimethylpentane	2.3	2.4	2.6	2.8	
2,3-Dimethylpentane	2.7	2.7	3.0	2.8	
Heptane	2.8	4.1	3.2	4.4	
Methylcyclohexane	2.1	2.8	2.4	2.9	
2,2,3,3-Tetramethylbutane				1.4	
Toluene				1.2	
2,3-Dimethylhexane	1.5	2.9	1.8	6.1	
3-Ethylhexane		1.9	1.3	4.0	
Octane	0.9	1.9	1.1	5.2	
2,4,4-Trimethylhexane				1.1	
Dimethylheptanes, 4,4/2,6	•			1.4	
1,4-Dimethylbenzene				2.4	
4-Methyloctane				2.6	
3-Methyloctane				1.1	
1,2-Dimethylbenzene				1.6	
Nonane		0.8	0.3	4.0	
Dimethyloctanes, 2,7/3,5/3,6				1.0	
3,3-Diethylhexane				1.1	
& Ethyloctane, 3/4					
Decane		0.3		1.9	
					
Total	92.1	85.3	87.8	72.1	

TABLE 102. VAPOR COMPOSITIONS OF JF-8, TANK F-3

		Weight	Percent	,
Component	32°F	100°F	135 °F	170°F
	,			
Butane	46.0	3.9	2.1	2.3
2-Methylbutane	18.3	1.1	-	-
1,1,2-Trimethylcyclopropane	1.3	•	-	, 1.7
Heptane	1.3	· · ·	-	-
3-Ethylhexane	1.1	~	<u>-</u> -	_
Octane	1.1	0.4	0.2	0.4
2,4-Dimethylheptane	1.4			
Nonane	2.3	1.0	1.1	1.4
2,3,4-Trimethylheptane	1.0	- · · · · · · · · · · · · · · · · · · ·	· 1.2	1.2
Trimethyloctanes,	• • • •	_	1.0	1.1
(2,2,6/2,2,7/2,4,4)				•
Decane	3.).	3.4	4.1	3.6
5-Ethylnonane	1.4	1.8	2.0	2.0
Dimethylnonanes, (3,5/3,6)		-,	1.1	1.0
3,3-Dimethylnonane	1.3	1.3	1.4	1.4
Trimethyloctanes,	1.0	1.5	1.6	1.6
(4,4,5/3,4,4/2,3,4)			•	
5-Methyldecane	_	1.1	1.2	1.2
3-Ethylnonane	-	1.0	1.1	1.1
2-Methyldecane	_	1.9	1.8	1.8
2,5,8-Trimethylnonane &	-	1.8	1.9	2.0
Dimethylethylbenzenes *	,			•
4,5-Diethyloctane	-	1.4	2.3	3.4
Hendecane & Methyl	4.4	11.1	10.6	8.2
Isobutylbenzenes **				
2,6-Dimethyldecane	-	3.0	2.9	3.1
3,6-Diethyloctane	_	2.6	2.6	2.7
Hexylcyclopentane	•	2.9	2.5	2.5
5-Methylhendecane		1.4	1.4	1.3
4-Cyclopentylheptane	-	1.4	1.3	1.2
3-Methylhendecane		2.3	2.3	2.2
1,6-Dimethylindan	· -	1.8	1.8	1.8
1,4-Diisopropylhexane		1.7	1.5	1.3
Hexamethylcyclohexane	-	.—	1.3	1.2
3,5-Dimethyl-5-Ethylnonane	-	1.1	1.1	1.0
4-Propyldecane	_	1.5	1.4	1.3
Dodecane	1.7	6.5	6.6	5.7
Butylcycloo ctane	-	2.2	2.0	1.7
Tridecane		1.8	1.6	1.6
Total	86.7	64.5	66.7	64.0

^{*} Weight ratio of 3:1
**Weight ratio of 10:1

may have had a low vapor pressure, made some contribution to the composition of the vapor. A total vapor analysis would consequently have included most of the compounds present in the fuel, particularly at temperatures above ambient. The portion of the vapor that is accounted for by the reported compounds is given as a summation at the bottom of each column in the tables.

It should be noted that values presented in the tables are relative vapor compositions. The concentration of any given component in the air head space could be estimated using these data along with appropriate vapor pressure values.

Discussion

As shown in Table 102, JP-8 vapors at 32°F consisted largely of the very volatile constituents, mainly butane (46%) and 2-methylbutane (18%). These components were present only as trace constituents of the fuel liquid phase, being scarcely perceptible in a chromatogram of the fuel. Replicate vapor analyses at 32°F, however, showed their preponderance in the vapor phase. As temperature was increased, the other JP-8 components contributed increasingly to the vapor composition. At 170°F, butane and 2-methylbutane constituted only a small part of the vapor. This observation emphasizes the need to consider the total amount of vapor (vapor pressure) as well as its composition in applying these data to specific problems.

In several cases, where components were chromatographically unresolved, approximate weight ratios were determined by mass spectrometry and these are reported in Table 102.

15. PARTIAL CHARACTERIZATION OF SHALE-DERIVED FUELS

Five samples of shale-derived fuel were characterized by simulated distillations, elemental analyses, and hydrocarbon-type analyses.

Simulated distillations were conducted by gas chromatography as described in ASTM Method D 2887. A gas chromatograph, equipped with a flame ionization detector and a column of 3% OV-1 on Chromosorb W, was used for this work. Data are presented in Table 103.

TABLE 103. SIMULATED DISTILLATION BY GAS CHROMATOGRAPHY

							,			•
Percent Recovery	X090	0-176 °F	X090	0-177 °F	<u>x090</u>	0-178 °F	<u>x09</u>	0-179 °F	<u>x090</u>	0-180 °F
0.5 (IBP)	51	124	116	241	28	83	27	81	88	190
1	59	138	120	248	34	93	29 29	84	92	198
5	77	171	140	284	64	147	60	140	120	248
10	92	198	156	312	83	181	79	174	137	279
· 20	116	241	174	345	98	208	110	230	161	322
30	131	268	188	370	115	2.39	142	288	181	358
40	144	291	198	388	123	253	170	338	199	390
50 ·	165	329	210	410	135	275	206	403	216	421
€0	185	365	218	424	146	295	222	432	231	449
70	206	403	225	437	164	327	235	455	249	480
80	226 /	439	236	457	183	361	252	486	266	511
90	244	471	253	487	212	414	280	536	281	538
95	257	495	269	516	230	446	. 301	574	290	554
99	299	570	288	550	249	480	324	615	309	588
99.5 (FBF)	308	586	291	556	254	489	328	622	312	594

Elemental analysis was obtained from a commercial microanalytical laboratory, Galbraith Laboratories, Inc., Knoxville, Tennessee. Data are presented in Table 104.

Hydrocarbon-type analyses were conducted by mass spectrometry using a modification of ASTM D 2729. Date are presented in Table 105.

TABLE 104. ELEMENTAL ANALYSIS OF SHALE-DERIVED FUELS

		Weight &							
Sample	Carbon		Hydrogen		Nitrogen				
X090-176	86.24 86.53		13.55 13.50		0.004				
	Mean	86.36	2014.0	13.53		0.004			
x090-177	85.99 86.19		13.79 13.68		0.006				
	Mean	86.09		13.74		0.006			
x090-178	85.14 84.96		14.66 14.43		0.002 0.002				
,	Mean	85.05		14.55		0.002			
X090-179	86.21 86.32		13.34 13.67		0.005 0.005				
	Mean	86.27		13.51		0.005			
x090-180	86.55 86.73		13.38 13.12		0.004 0.004				
	Mean	86.64		13.25		0.004			

TABLE 105. HYDROCARBON-TYPE ANALYSIS BY MASS SPECTROMETRY

		Volume %					
Components	X090-176	X090-177	X090-178	X090-179	X090-180		
Paraffins	46.7	46.4	55.2	46.0	34.0		
Monocycloparaffins	39.6	40.3	35.8	40.7	46.0		
Dicycloparaffins	-	-	2.1	-	3.2		
Alkylbenzenes	9.5	6.8	5.5	7.3	9.2		
Indars & Tetralins	4.2	6.0	0.9	5.7	6.9		
Naphthalenes	Trace	0.5	0.5	0.3	0.7		

⁻ Dash indicates none found.

16. DIELECTRIC CONSTANT OF A JP-4 SAMPLE LABELED KI SAWYER

Dielectric constant was measured at three temperatures for KI Sawyer JP-4 sample #80-37, Tank 5, drawn 14 November 1980. The measurements were made relative to air at 400 hertz using a three-terminal guarded cell and a General Radio 1615A capacitance bridge and guarded circuit. The bridge signal generator was a General Radio 1311A audio oscillator, and the bridge detector was a Type 1232A tuned amplifier and null detector. The test results are listed below.

Test temperature, °F	Dielectric constant
32	2.102
77	2.052
100	2.028

17. COMPARATIVE CARBON-HYDROGEN ANALYSES OF EIGHT FUEL SAMPLES BY TWO COMMERCIAL LABORATORIES

Six samples comprising a mixture of actual jet fuels and synthetic fuel blends were submitted to two commercial analytical laboratories for duplicate carbon and hydrogen analyses. The two commercial companies were Galbraith Laboratories, Inc., and Schwartzkopf Microanalytical Laboratory, both of which use the classical combustion technique for their analytical approach. The analyses were required as part of a program to evaluate different methods for measuring hydrogen content. Two of the six samples were resubmitted six weeks later to the same two laboratories as blind duplicates for a measure of method reproducibility. All data are presented in Table 106.

TABLE 106. DUPLICATE CARBON AND HYDROGEN ANALYSIS RESULTS FROM TWO COMMERCIAL LABORATORIES FOR JET FUELS

Sample	Replicate			bon Data From	Percent Hydr	ogen Data From
Number	Number		Galbraith	Schwarzkopf	Galbraith	Schwarzkopf
80-304-1	1		87.69	87.50	12.35	12,68
00 301 1	2		87.50	87.48	12.47	12.59
	_	N				
		Avg.	87.60	87.49	12.41	12.64
80-304-2	ı		88.26	87.9 0	11.72	11.95
	2		88.25	88.10	11.80	11.87
		Avg.	88.26	88.00	11.76	11.91
80-304-3	1		87.63	87.55	12.38	12.45
	2		87.68	87.54	12.37	12.70
		Avg.	87.66	87.55	12,38	12.58
				07.55	12,50	12.50
80-304-4	1		88.00	87.97	12.07	11.79
	2		88.03	00.89	1' 94	11.67
		Avg.	88.02	87.99	12.01	11.73
80-304-5	1		86.30	85.58	13.68	14.57
	2		86.34	85.36	13.88	14.48
		Avg.	86.32	85.47	13.78	14.53
80-304-5	1		85.54	85.75	14.35	14.31
Blind Repeat	2		85.70	85.52	14.23	14.52
6 wks. later		Avg.	85.62	85.64	14.29	14.42
					24,23	44.44
80-304-6	1		86.54	86.81	12.89	13.05
(DF-2)	2		86.59	86.92	12.68	13.04
		Avg.	. 86.57	86.87	12.79	13.05
80-304-6	1		87.20	86.96	12.79	12.96
Blind Repeat	2		87.24	86.90	12.70	13.17
6 wks. later	•	Avg.	87.22	86.93	12.75	13.07

Conclusions

- 1. With the exception of Sample 80-304-4, Galbraith Laboratories consistently obtained lower hydrogen values than Schwartzkopf.
- 2. The Schwartzkopf percent hydrogen data appeared to be in good agreement with data obtained on similar samples analyzed at APL by an NMR technique.
- 3. Good reproducibility was obtained by Schwartzkopf Laboratories for the blind repeat analyses of both diesel fuel and JP-4. Good reproducibility was also obtained by Galbraith Laboratories for the diesel fuel analyses, but the dissimilar results for the JP-4 fuel analyses indicates that Galbraith may have problems in handling volatile samples.

It should be noted that both of the laboratories quote an absolute accuracy of ±0.3% for carbon and hydrogen determinations. Analysis from the two laboratories do not differ by more than this amount for any sample except sample 80-304-5.

18. EFFECT OF ANTISTATIC ADDITIVES ON THE DIELECTRIC CONSTANT OF JP-4 FUEL

Dielectric constant values were determined for a specimen of JP-4 fuel coded 81-3-CRM and separated portions of that fuel containing either 2 ppm ASA-3 additive or 2 ppm Stadis 450 additive. The purpose of the analyses was to determine if the presence of antistatic additives had a significant impact on the dielectric constant of the fuel, thereby affecting the accuracy of aircraft fuel gauges. This same fuel and additives were also evaluated for electrical conductivity and charging tendency as documented in subsection II-2. The dielectric constant values are listed below.

Test sample	Dielectric constant at 77°F
81-3-CRM JP-4 Fuel	2.046
JP-4 + 2.0 ppm ASA-3	2.031
JP-4 + 2.0 ppm Stadis 450	2.021

The above variations in dielectric constant are not any greater than would be expected for different lots of JP-4 without additives.

19. HYDROCARBON-TYPE ANALYSES OF JP-5 AND JP-8

Nine samples of JP-5, JP-8, and shale-derived broad range fuels were analyzed by mass spectrometry to determine their hydrocarbon-type distributions. The modified ASTM D 2789 method and Monsanto Method 21-PQ-38-63 were used for these determinations. Average density values were used to convert ASTM D 2789 volume percent data (Table 107) to weight percent. The converted values are compared with those obtained by the Monsanto Method in Table 108.

TABLE 107. HYDROCARBON-TYPE ANALYSIS BY MODIFIED ASTM D 2739

				Volu	me Perc	ent			
	VN-81	VN-81	VN-81	VN-81	VN-81	VN-81	VN-8.1	VN-81	VN-81
Compound Type	-132	-133	-134	-135	136	<u>-137</u>	<u>-138</u>	-139	140
Paraffins	49.6	50.1	49.8	39.5	35.5	40.9	42.1	50.2	48.6
Cycloparaffins	38.7	33.0	34.5	40.1	51.0	42.8	32.5	20.7	28.3
Dicyclo- paraffins	0.0	0.0	0.0	12.9	3.4	9.2	4.3	0.0	0.9
Alkylbenzenes	5.5	8.1	7.7	4.0	6.8	3.7	10.3	17.6	13.9
Indans & Tetralins	5.6	7.2	6.6	3.1	3.1	3.0	9.8	10.7	7.7
Naphthalenes	Ů.6	1.6	1.4	0.4	0.4	0.4	1.0	0.8	0.6

TABLE 108. COMPARISON OF HYDROCARBON-TYPE ANALYSES

								_	feight 7	ercent								
	1	-132	=	-133	13	12.	8	-135	1-13	2	1-18	37		86	-18	661	1-10	3
	02789	TON ST	D2789	FORE	D2789 NOMS ^D D2789 NOMS D2789 NOMS D2789 NOMS D2789 NOMS D2789 NOMS D2789 NOMS D2789 NOMS	HOME	D2789	HOH	0270	HOMS	05789	FORES	02789	XON .	02789	MON	S 02789 NORS	
Paceffins	47.0	69.0	47.3	49.5	47.0	49.1	37.3	36.7	33.4	33.0	38.7	36.3	39.3	39.9	4,4	45.5	45.6	£.3
Cycloparaffins	39.6	39.6 36.9°	33.5 29.7	29.7	35.1	31.6	41.0	35,1 31.6 41.0 54.8	51.8 54.8	54.8	43.8 53.6	53.6	32.7 35.3	35.3	20.	20.8 18.0	28.6 27.6	27.6
Dicycloparaffins	,		1		•		13.1				9.5		4.3		•		6.0	
Alxylbenzenes	6.0	7.5	9.	11.5	4.4	10.7	4.4	4.9	7.2 8.5	.5	4.0	1 4.4 1	11.2	11.2 13.9	19.0	24.7	15.1	19.3
Indans & Tetralins	9.9	5.7	:	9.	7.7	9	3 3.7 3.0	3.0	3.6	3.3	3.5	3.1	11.3 9.5	9.5	12.4	10.9	9.0	7.9
Indenes & DAN	v	•		0.1		0.1		•				,				ŀ		•
Maphthalenes	6	6.0	2.0	2.0 2.4	1.8		0.5	2.2 0.5 0.6	0.5	3.6	3.4 0.5	9.6	1.2	1.4	1,0	0.6 1.2 1.4 1.0 0.9	0.8 0.9	6.0

A Value from modified ASTM D 2789 was converted from volume percent using average density values.

b Moncanto method 21-70-38-63.

c Total cycloparaffins.

d Method does not provide this information.

e Dash indicates none was detected.

20. HYDROCARBON-TYPE ANALYSES OF SHALZ-DERIVED GASOLINE, JP-4, JP-8, AND DF-2 BY TWO MASS SPECTRAL METHODS

Six shale-derived fuel samples consisting of two diesel fuels, two JP-8s, a JP-4, and a gasoline were analyzed by mass spectrometry to determine hydrocarbon-type distributions. The modified ASTM D 2789 method and Monsanto Method 21-PQ 38-63 were used for these determinations. The Monsanto method was used for all fuels because it was specifically requested although that procedure is not very appropriate for two of the samples. Samples VN 81-J41 and VN 81-142 are shale-derived JP-4 and gasoline, respectively. Their average carbon numbers of 8.4 and 8.6, respectively, place them outside the range intended for this method. The ASTM D 2789 data are considered to be the more reliable for these two samples.

Average density values were used to convert ASTM D 2789 volume percent data (Table 109) to weight percents. The converted values are compared with those obtained by the Monsanto method in Table 110.

TABLE 109. HYDROCARBON-TYPE ANALYSES BY MODIFIED ASTM D 2789

			Volume P	ercent		
Compound Type	81-141	81-142	81-143	81-144	81-145	81-146
Paraffins	43.4	43.2	50.7	48.6	39.8	40.4
Cycloparaffins	35.5	6.0	28.6	29.3	37.3	27.8
Dicyloparaffins	9.0	0.3	2.7	1.0	5.6	1.8
Alkylbenzenes	9.6	42.5	4.4	5.2	9.6	14.7
Indans & Tetralenes	2.0	6.9	11.7	13.8	6.9	14.0
Naphthaleres	0.5	1.1	1.9	2.1	8.0	1.3

TABLE 110. COMPARISON OF HYDROCARBON-TYPE ANALYSES

60

	46	3	36.3	20.1	0.5
	81-146	002200	37.2	15.7	1.6
	53	1 5	1 10 00	12.5	0.1
	81-145	02789	37.3	10.5	1.0
	44	MOM		7.1	1.6
Weight percent	81-144	D2789		5.6 15.9	2.6
ight p	43	MON	49.2	6.0	1.7
¥	81-143	D2789	47.5 29.0	4.8 13.5	2.4
	42	MOM	29.6 3.3	59.8 6.5	0.8
	81-142	02789	39.5 6.0 0.2	45.1 7.8	1.4
	41	MON	32.1 50.2	14.9 2.8	⁴⁴ , ,
	81-14	D2789	41.0 36.2 9.2	10.6 2.4	0.6
	Hydrocarbon	type	Paraffins Cycloparaffins Dicycloparaffins	Alkylbenzenes Indans and tetralins	Indenes and DHN ^G Naphthalenes

avalues from modified ASTM D 2789 were converted from volume percent using average density values. b Monsanto method.

crotal cycloparaffins.

d DHN = dihyáronaphthalenes.

Method does not provide this information.

 $f_{
m Dash}$ indicates none detect.d.

21. HYDROCARBON-TYPE ANALYSES OF SHALE-DERIVED JP-4, JP-5, JP-8, DF-2, AND DFM BY TWO MASS SPECTRAL METHODS

Nine shale-derived fuels including marine diesel fuel, DF-2, JP-4, JP-5, and JP-8; and two blend stock samples were analyzed for hydrocarbon types by two mass spectral methods. Analysis results from ASTM Method D 2789 (modified), in volume percents, are presented in Table 111. The same analysis results converted to weight percents and weight percent data from Monsanto Method 21-PQ-38-63 are presented in Table 112 for direct comparison.

The three JP-4 fuels in this set of samples had average carbon numbers, as determined by mass spectrometry, of 9.6 for 81-149; 7.9 for 81-150 and 8.5 for 81-155. The average carbon number for the latter two fuels placed them outside the intended range of the Monsanto method, thus the ASTM Method D 2789 (modified) data were considered more reliable. However, both sets of values were reported as specifically requested.

22. HYDROCARBON-TYPE ANALYSES BY TWO MASS SPECTRAL METHODS FOR TEN SHALE-DERIVED JP-4 AND JP-8 FUELS; SIMULATED DISTILLATIONS FOR TWO FUELS

Ten shale-derived JP-4 and JP-8 fuel samples were analyzed for hydrocarbon types by two mass spectral methods, ASTM D 2789 and Monsanto Method 21-PQ-38-63. Analytical data from ASTM D 2789, in volume percents are presented in Table 113. Weight percent data from both the ASTM and Monsanto methods are presented in Table 114 for direct comparison.

Two of the JP-4 samples, WEH-81-37 and VN-81-158, were also analyzed for boiling range distributions via simulated distillations by gas chromatography (ASTM D 2887). These results are provided in Table 115.

HYDROCARBON-TYPE ANALYSES BY ASTM D 2789 - VOLUME PERCENTS

Indans & Maphtha- Tetralins lenes								
20 1	4.7							
Dicyclo- Paraffins	0.0	3.6	1.9	0.0	1.3	0.4	3.7	
Monocyclo- Paraffins	41.0	36.4	37.0	39.5	36.1	34.6	37.3	
Paraffins	51.0	50.8	37.7	37.2	46.8	53.9	53.5	
Scurce	Shale	Shale	Shale	Shale	Shale	Shale	Shale	
Fue1 Type	JP-4	JP-4	JP-5	JP-8	DF-2	Marine-DF	JP-4	
		C. 1-1.0		21-10-	e91-153	M-81-154	81-155	

HYDRCCARBON-TYPE DATA FROM TWO MASS SPECTRAL METHODS - WEIGHT PERCENTS TABLE 112.

THE REPORT SHOULD SELECTE SELECTED SELECTED SERVICES SELECTED SERVICES SELECTED SELE

5				44.1 44.8 51.3 52.6						WEH-81-40	Blended Stock	D2789 MONS	42.6 41.0		9.7 7.8	
6	VN-81-152	Shale JF-8	D2789 MONS	34.5 34.2	39.6 38.2	14.3 18.2	10.5 8.2	0.0	F. 1 1.2	WEH-81-39	Blended Stock	D2789 MONS	15.7 12.8		5.2 5.1	
	VN-81-151	Shale JP-5	D2789 MONS	34.4 35.1	38.2 38.2	14.1 16.0	12.1 9.4	0.0	1.2 1.3	VN-81-157	Shale JP-5	D2789 MONS	40.9 41.2			
i i	VN-81-150	Share JP-4	D2789 MONS	48.4 41.0	41.2 45.2	6.8 10.3	2.8 3.5	0.0	0.8 0.0	VN-81-155	Shale JP-8	D2789 MONS			5.2 4.3	
3	VN-81-149	-1	D2789a MONSD	48.6 51.9	42.2 37.4	5.2 6.8	3.7 3.4	0.0	0.3 0.5	VN-81-155	6	D2789 MONS	51.3 45.8	42.4 45.2	1.3 1.6	
		Components		Paraffins	Cycloparaffins	Alkylbenzenes	Indans/Tetralins	Indenes ^C	Naphthalenes			Components	Paraffins	Cycloparaffins	Indans/Tetralins	ָרָ

a ASTM Method D2789 provides data in volume percents (see Table 1). The weight percent data were calculated from average density values for the compound classes.

b Monsanto mass spectral method 21-PQ-38-63 was developed for hydrocarbon fuel stocks.

^C Also includes dihydronaphthalenes in the analysis. This compound class is not detected by ASTM D2789.

HYDROCARBON-TYPE ANALYSES BY ASTM D 2789 (MODIFIED) - VOLUME PERCENTS Naphtha-Tetralins Indans & 2.3 4.1 Benzenes Alky1 3.5 11.5 10.4 Paraffins Dicyclo-0.0 3.9 0.0 0.0 Monocyclo-Paraffins 29.3 43.3 46.9 26.9 53.5 41.2 42.4 28.8 35.3 33.7 Paraffins 44.8 48.3 39.5 47.4 51.6 61.9 47.6 58.7 Shale Shale Shale Shale Shale Shale Shale Shale Shale Shale Source Type JP-4 JP-4 JP-4 JP-4 JP-4 JP-4 JP-8 JP-4 JP-4 JP-8 Fuel TABLE 113. VN-81-125 VN-81-126 VN-81-158 VN-81-159 VN-81-122 VN-81-123 VN-81-124 WEH-81-37 VN-81-120 VN-81-121 Sample Number

the state of the s

HYDROCARB')N-TYPE DATA FROM TWO MASS SPECTRAL METHODS - WEIGHT PERCENT TABLE 114.

	VN-81-120	-120	VN-81-121	-121	VN-81-122	122	VN-81-123	123	VN-81-124	124
Components	Shale JP-4 D2789a MON	JP-4 MONSD	Shale JP-4 D2789 MON	JP-4 MONS	Shale JP-4 D2789 MON	JP-4 MONS	Shale JP-4 D2789 MON	JP-4 MONS	Shale JP-4 D2789 MON	MONS
	42.7	34.5	37.4	38.2	45.4	37.3	49.2	52.3	59.5	56.2
Farattins	50.8	57.5	54.9	53.7	52.5	60.2	42.5	38.4	32.1	33.2
Cyclopararrins	5.0	7.1	3.7	4.0	1.4	2.2	3.9	4. 9	4.9	7.4
Alkylbenzenes	0.9	6.0	3.5	3.4	0.2	0.3	4.0	3.8	2.7	3.2
Indans/Tetralins	1	0.0		0.0	1	0.0	1	0.0	ı	0.0
Indenes ^C	9.0	0.0	0.5	0.7	0.5	0.0	0.4	9.0	0.8	0.0
Naphiniatenes	8	. 3	10	10.3	w	8.0	6	80	ω	9.8
averaye carbon no.										
	VN-81-125	-125	VN-81-126	-126	VN-81-158	.158	VII-81-159	159	WEH-81-37	37
	Shale	JP-8	Shale JP-4	JP-4	Shale JP-4	JP-4	Shale JP-8	JP-8	Shale JP-4	JP-4
Components	D2789 MONS	MONS	D2789	MONS	D2789	MONS	D2789	MONS	D2789	MONS
Paraffins	45.8	47.8	56.4	53.3	44.9	42.0	44.6	46.3	47.6	42.7
Cycloparaffins	43.5	41.0	36.1	36.5	35.9	34.2	34.2	31.0	34.8	33.9
Alkylbenzenes	5.1	0.9	4.7	7.0	12.6	17.4	11.3	14.4	11.7	17.0
Indans/Tetralins	4.8	4.2	2.4	2.9	6.1	5.9	0.6	7.0	5.3	5.8
Indenesc	ı	0.0	ı	0.0	1	0.0	i	0.0	ı	0.0
Naphthalenes	0.8	1.0	0.4	0.3	0.5	0.5	6.0	1.3	9.0	9.0

ASTM Method D2789 provides data in volume percents (see Table 113). The weight percent data were calculated from average density values for the compound classes.

Average Carbon No.

b Monsanto mass spectral Method 21-PQ-38-63 was developed for hydrocarbon feed stocks.

CAlso includes dihydronaphthalenes in the analysis. This compound class is not detected by ASTM D 2789.

TABLE 115. SIMULATED DISTILLATION BY GC (ASTM D 2887)

			2	(;))
Percent	WEH-81-37 Shale JP-4	1-37 JP-4	VN-81 Shale	VN-81-158 hale JP-4
Recovered	ပ်ျ	<mark>(</mark>	ပ္သ	40
0.5 (IBP)	- 26	79	34	93
rH	34	93	26	133
5	69	156	75	167
10	91	196	76	207
20	118	244	121	250
30	140	284	142	288
40	161	322	164	327
50	181	358	183	361
09	202	396	202	396
70	220	428	221	430
80	236	457	236	457
06	257	495	257	495
95	269	516	269	516
66	293	559	292	558
99.5 (FBP)	300	572	299	570

The average carbon numbers for all JP-4 fuels were calculated and are presented in Table 114. Samples 81-120, 81-122, and 81-124 had carbon numbers outside the intended range of the Monsanto method, thus the ASTM D 2789 data were more reliable for these samples. However, both sets of data were reported as requested.

23. HYDROCARBON-TYPE ANALYSES OF FIVE SHALE-DERIVED JF-8, DF-2, AND DFM SAMPLES BY TWO MASS SPECTRAL METHODS

Five shale-derived fuel samples were analyzed for hydrocarbon-type distribution by two mass spectral methods, modified ASTM Method D 2789 and Monsanto Method 21-PQ-38-63. The data from ASTM Method D 2789 are provided in Table 116 in volume percents. The same data were converted to weight percent values by the use of average densities for the compound classes, and these values are presented in Table 117 along with the weight percent values from Monsanto Method 21-PQ-38-63.

TABLE 116. HYDROCARBON-TYPE ANALYSES BY MODIFIED ASTM D 2789 - VOLUME PERCENTS

Compound Type	Shale Marine Diesel Fuel VN-81-160	Shale DF-2 VN-81-161	Shale Broad Range JP-8 VN-81-162	Shale Broad Range JP-8 VN-81-163	Shale JP-8 VN-81-164
Paraffins	48.1	48.0	46.1	46.9	50.5
Cycloparaffins	38.3	38.6	39.9	40.2	41.4
Dicycloparaffins	0.9	0.7	0.2	0.0	0.0
Alkylbenzenes	5.3	5.3	5.8	5.6	4.0
Indans/Tetralins	6.0	6.1	6.7	6.1	3.5
Naphthalenes	1.4	1.3	1.3	1.2	0.6

24. DENSITY OF 14 MULTI-TYPE SHALE-DERIVED FUELS AT 15°C

Density was determined by the dilatometer method for 14 shalederived fuels at 15°C. Results are shown in Table 118.

HYDROCARBON-TYPE DATA FROM TWO MASS SPECTRAL METHODS - WEIGHT PERCENTS TABLE 117.

	Shale M Diesel VN-81-	Shale Marine Diesel Fuel VN-81-160	Shale D7-2 VN-81-161	D?-2 -161	Shale Range VN-81	Shale Broad Range JP-8 VN-81-162	Shale Broad Range JP-8	Broad JP-8	Shale JP-8	JP-8
Components	D2789a MONSD	GSNOW	D2789 MONS	MONS	D2789	MONS	D2789	SNOW	D2789 MON	WONS
Paraffins	45.4	46.3	45.3	46.1	43.4	44.5	44.3	45.2	48.1	5.0.5
Cycloparaffins	39.9	38.4	40.1		40.8	39.0	41.0	39.0	42.6	39.6
Alkylbenzenes	5.8	6.7	5.8	8.0	6.4	7.5	6.1	7.3	4.4	5.2
Indans/Tetralins	7.1	5.6	7.1	5.7	7.8	6.3	7.1	5.8	4.1	3.6
${\tt Indenes}^{\scriptscriptstyle\mathcal{C}}$	ı	6.0	ı	6.0	ı	1.0	i	6.0		0.0
Naphthalenes	1,8	2.1	1.7	2.0	1.6	1.7	1.5	1.8	0.8	1.0

These the compound ^aASTM Method D 2789 (Modified) provides data in volume percents (see Table 116). weight percent data were calculated from average density values for the compound classes.

b Monsanto mass spectral Method 21-PQ-38-63.

This compound class is not detected $^{\mathsf{C}}\mathsf{Also}$ includes dihydronaphthalenes in the analysis. by ASTM D 2789.

TABLE 118. DENSITY OF SHALE FUELS AT 15°C

Sample number	Type fuel	Density, g/cc
VN-81-141	Shale JP-4	0.7825
VN-81-142		
· · · · · · · · · · · · · · · · · · ·	Shale gasoline	0.8034
VN-81-143	Shale DF-2	0.8345
VN-81-144	Shale DF-2	0.8359
VN-81-145	Shale JP-8	0.8178
VN-81-146	Shale broad range	0.8269
	JP-8	
VN-81-149	Shale JP-4	0.7801
VN-81-158	Shale JP-4	0.7871
VN-81-159	Shale JP-8	0.8094
VN-81-160	Shale DFM	0.8308
VN-81-161	Shale DF-2	0.8307
VN-81- 162	Shale JP-8 blend	0.8205
VN-81-163	Shale JP-4 blend	0.8217
VN-81-164	Shale JP-8	0.7969

25. DENSITY AND BOILING RANGE DISTRIBUTION FOR EIGHT BP-IP CODED FUELS

Eight coded fuel samples were evaluated for density at 15°C and boiling range distribution. Density was determined by the dilatometer method and results are presented in Table 119. Simulated distillations were conducted by gas chromatography according to ASTM Method D 2887, and these results are provided in Table 120.

TABLE 119. DENSITY OF FUEL SAMPLES

Sample code	Grams/cc
BP-IP-1	0.7989
BP-1P-2	0.7888
BP-IP-3	0.7948
BP-IP-4	0.8002
BP-IP-5	0.7990
BP-IP-6	0.8293
BP-IP-7	0.8180
BP-IP-9	0.8309

TABLE 120. SIMULATED DISTILLATIONS

4-41	253	363	396	437	4 4 6	513	BP-IP-9	217	338	385 399 159	432	484 496 496	523 532
BP-IP-4	123	184	202 210 210	233 233 233	242 251 256	267 269	E C	103 116 154	170 185	196 204 213	232	7 2 3 3 4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	273 278
BP-IP-3	212 239 282	300	345 365 85	4 28	1 6 6 1 0 6 1 0 6	534 554	7-4 0-1	214 230 297	327	372 387 4 05	423 441 457	495 495	523 538
da o	100	149	174 185 196	200	24 9 2 2 2 8 9 3 2 9 3 9 3 9 9 9 9 9 9 9 9 9 9 9 9 9	279 290	BP-IP-7	101	164	189 197 207	217 227 356	250	273 281
7 PH	243 262 298	309 327	342 354 369		118 173	516 541	اه بو	216 243 307	338	387 403 419	133	486 498	520 525
BP-IP-2			172		231		BP-IP-6				223		271
[P-1	255 280 336	356 381	396 412 423	437	486 496 496	520 538	16-5 - P	237 246 279	295	325 340 352	376	462	\$11 \$20
3P-IP-1	124 138 169	180	202 211 217	233	7 2 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	271 281	BP-IP-5	114 119 137	146	163 171 178	191 204 221	233 233 223 223	266 271
Percent Recovered	0.5 (I3P) 1 5	10 20	04 0 04 0 	2000	0 6 6 0 6 6	99 99.5 (FBP)		0.5 (IBP) 1 5	10 20	50 4 50 50 6	60 70 80	90	99 99.5 (FBP)

26. ICP TRACE METALS ANALYSES FOR TEN SHALE- AND PETROLEUM-DERIVED JP-4 AND JP-8 FUELS

Two samples of JP-4 and JP-8 tuels derived from both shale and petroleum sources were analyzed for trace metals content using an ISA Model JY48P inductively coupled plasma (ICP) spectrometer. The ICP analyses were conducted on aqueous acid (ultrapure HCl) extracts of the fuels which provided a concentration factor of 21.25 for the metals. The test results for an acid extraction blank, the fuel samples (one in duplicate), and a reference standard are shown in Table 121.

27. ICP TRACE METALS ANALYSES FOR THREE JP-4 FUELS

Three fuel samples were analyzed for trace metals content by inductively coupled argon plasma (ICP) spectroscopy. The instrument used was an ISA Model JY48P ICP spectrometer. The analyses were conducted on aqueous acid extracts of the fuels which were obtained using ultrapure hydrochloric acid. The concentration factor for metals in the acid extract was 21.25. The analytical results for an acid extraction Liank, the three fuel samples and a reference standard are presented in Table 122.

Though sodium is not always included in ICP analyses, the instrument was configured to include sodium for these analyses. Because of the ubiquitous nature of sodium and the fuel's history of contact to soft glass, the analysis for sodium is not considered to have the same significance as the analyses for other metals, and it was thus excluded from the table. As these values could have served to monitor gross sodium contamination, they were provided in a separate listing as follows.

TABLE 121. TRACE METALS ANALYSES BY ICP SPECTROMETRY

CANAL SECRETARY

The state of the s

8	270	13	×		×	×	×	×	×	×	×	×	×	×	×		1000 1000	1040	101
욅	125	•	×		×	×	×	×	×	×	×	×	×	Ħ	×		0001	\$	\$
8	22	~	×		×	×	×	×	×	×	×	×	×	M	×		1000	1000	100
2	91	9.5	×		1.7	1.5	1.7	1.7	1.7	1.5	1,4	1.6	1.8	1.9	1,5		1000	1030	103
5	•	0.4	×		0.7	×	1.1	1.6	×	*	1.6	1.1	3.6	3.6	×		1000	1030	103
£	175	8.2	×		×	×	×	×	×	×	×	×	×	×	43		1000	1000	100
되	120	5.7	×		×	×	×	×	×	×	×	×	×	*	×		1000	1100 1000	110
히	22	-	×		×	×	×	×	×	×	×	×	×	×	×		1000	1020	102
ھ ا	35	1.6	×		3.0	×	×	×	2.3	×	7.0	×	×	×	1.7		1000	1110	111
T	0 0	0.4	×		×	×	×	×	×	×	×	×	×	×	×		1000	1020	102
Ä	86	•	×		×	×	×	×	×	×	×	×	×	×	×		1000	986	\$
2	9	0.5	×		20	3.9	3.6	5,9	6.3	2.0	8.0	8.0	1.8	2.1	3.6		1000	1040	104
밁	4	0.2	×		×	×	×	×	×	×	×	×	×	×	×			955 1020	102
7	150	7	×		×	×	×	×	×	×	×	×	×	×	×		1000 1000	955	96
S	250	12	×		×	×	×	×	×	×	×	×	21	61	×		١	×	
된	m	0.2	×		2,4	×	×	×	0.3	×	0.2	0.2	×	×	0.2		1000	1040	104
ଥ	24	1.1	×		×	×	×	×	×	×	×	×	×	×	×		1000 1000	1070 1040	107
ᆈ	7	0.1	×	6	×	×	×	×	×	×	×	×	×	×	×		1000	1010	101
হা	110	'n	×	Samples,	×	×	×	×	×	×	×	×	×	×	×		1	×	
Elements of Detection:	Instrument detection limits, ppb	MDL after concentration, ppb	Acid Extraction Blank	Elements Concentration in Fuel Sam	UOP-3794-157C	FOSF-D-81-124	-117	99 -	19 -	- 63	-114	- 43	17 -	Repeat - 44	JP-4-5-05288-1	Analysis of Reference Standard, ppb	True value	Observed Value	* Recovery

a The fuel samples were effectively concentrated by a factor of 21.25 when they were extracted with a much smaller volume of agueous acid for the analysis.

The aqueous ultrapure acid used to extract the fuel samples for analysis. An "x" shows that the element was not detected at the instrument detection limit

^C An "x" shows that the element was not detected at the MLL after concentration. The numerical values were obtained by dividing the observed value by 21,25 to take into account the concentration effect, and these values represent the element concentration in the original fuels.

TRACE METALS ANALYSES BY ICP SPECTROMETRY TABLE 122.

AND THE PROPERTY OF THE PROPER

>1	8	2.8			* * *
Sr	ν,	0.3	×		* * *
3	140	9.9	×		* * *
æl	270	13	ĸ		* * *
£İ	125	w	×		* * *
ઢા	22	-	×		4.7 7.3 1.6
2	70		×		0.8
5	6		×		×× 9.
21			×		* * *
되	120	5.7	×		* * *
비	22	1 5.7	×		× × ×
mi	35	9.1	×		***
리	80	0.4	×		* * *
=	96	4	×		* * *
21	10	0.5	×		* * *
핑	4	0.2	×		* * *
짇	150	7 0	×		7.1 × 7.6
S	250	12	×		* * *
된		1.1 0.2 12	×		* * *
ଥା	24	1.1	×	-1	* * *
2	~	5 0.1	×	304	×××
হা	110	'n	×	ples,	* * *
Elements of Detection:	Instrument detection limits, ppb 110	MDL after concentration, ppb	Acid Extraction Blank x x	Elements Concentration in Fuel Samples, ppb	JP-4-103081-3 JP-4-103021-4 VN-81-119

Analysis of Reference Standard, ppb

0	0	-	
1000	1010	101	
1000	1020	102	
1000	1020	102	
0001	972	4	
1000	1040	701	
1000	984	88	
1000	001	8	
1000	1050	105	
1000	1070	101	
1001	1030	103	
10001	978	86	
10001	1090	109	
1000	1010	101	
1000	066	66	
10001	1020	102	
1000	1040	104	
1000	965	16	
10001		106	
0001	0501	105	
•	1090	109	
1000 1000	1030	103	
0001	991 1030	66	
True Value 1	Observed Value	& Recovery	

a The fuel samples were effectively concentrated by a factor of 21.25 when they were extracted with a much smaller volume of aqueous acid for the analysis.

The aqueous ultrapure anid used to extract the fuel samples for analysis. An "x" shows that the element was not detected at the instrument detection limit

C An "x" shows that the element was not detected at the MDL after concentration. The numerical values were obtained by dividing the observed value by 21.25 to take into account the concentration effect, and these values represent the element concentration in the original fuels. The numerical values were obtained by dividing

Sample	concentration,
Blanks	140
JP-4 103081-3	640
JP-4 103081-4	730

VN 81-119

aBlank value has been subtracted from those of fuels.

SECTION IV

COMBUSTION SUPPORT

Certain Air Force programs require the determination of data describing the detailed chemistry of fuels being used in combustion studies. As a class, aromatic compounds are of greatest interest because they tend to have the poorest combustion characteristics. High concentrations of aromatics have been known to produce excessive exhaust emissions in the form of unburned hydrocarbons and smoke. Paraffins and cyclic paraffins, on the other hand, are clean burning and have a high gravimetric heat of combustion. Accurate compositional data are therefore often required as an integral part of fuel combustion studies.

The test programs described in this section have involved a wide range of fuel physical property and analytical determinations to complement the information being generated by the Air Force in separate fuel combustion studies.

1. CHEMICAL AND PHYSICAL PROPERTIES OF SIX CONVENTIONAL AND SYNTHETIC FUELS

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Six fuels of widely varying types, including those of conventional and synthetic origin, were subjected to several physical and chemical property tests using procedures detailed in the Appendix. Results for density and vapor pressure as a function of temperature are presented in Tables 123 and 124; net heat of combustion values by oxygen bomb calorimetry are shown in Table 125; results for simulated distillation by gas chromatography are presented in Table 126; and data for hydrocarbon type analysis by mass spectrometry are tabulated in Table 127.

TABLE 123. DENSITY OF TEST FUELS AS A FUNCTION OF TEMPERATURE

	De	ensity, g/co	
Sample	32°F	70°F	100°F
Conventional JP-4	0.7649	0.7484	0.7353
Shale JP-4	0.7633 0.8226	0.7466 0.8076	0.7332 0.7955
Conventional JP-5 Shale JP-5	0.8226	0.8024	0.7903
JP-9 (6-22-78) JP-10 (6-22-78)	0.9588 0.9516	0.9421 0.9355	0.9289 0.9230

TABLE 124. VAPOR PRESSURE OF TEST FUELS AS A FUNCTION OF TEMPERATURE

	Vaj	or Pressi	ure,
Sample	32°F	70°F	100°F
Conventional JP-4	28.5	67.0	122.0
Shale JP-4	24.0	55.5	99.5
Conventional JP-5	10.8	15.7	20.3
Shale JP-5	6.6	11.0	15.7
JP-9 (6-22-78)	7.5	17.5	29.0
JP-10 (6-22-78)	4.6	8.6	13.5

TABLE 125. HEAT OF COMBUSTION OF TEST FUELS (ASTM D 240-64)

The second secon	to a some maximum of the manufacture of the same of th		
	Gross,	Btu/lb	Net, Btu/lb
Sample	Duplicate	Average	Average
Conventional JP-4	20,099	1 .	
,	20,098	!	
	• •	20,059	18,768
Shale JP-4	20,091	• .	•
	20,102		
•		20,097	18,772
Conventional JP-5	19 92		
	19,777		
	-	19,785	18,511
Shale JP-5	19,749		
	19,748		
	·	19,749	18,497
JP-9 (6-22-78)	19,098		
	19,071		
		19,085	18,020
JP-10 (6-22-78)	19,146		
- •-	19,163		
		19,155	18,074

ACCOUNTS OF THE PROPERTY OF TH

TABLE 126. GAS CHROMATOGRAPHIC SIMULATED DISTILLATION OF TEST FUELS

Percent Recovered	°C	°F	• C	°F
	Convention	al JP-4	Shale J	P-4
0.5 (IBP) 1 5 10 20 30 40 50 60 70 80 90 95	4 22 58 79 96 115 135 158 181 202 217 236 251 269	39 72 136 174 205 239 275 316 358 396 423 457 484 516	2 22 70 90 107 122 136 148 161 174 187 202 214 241	36 72 158 194 225 252 277 298 322 345 369 396 417 466
99.5 (FBP)	277	531	260	500
	Convention	al JP-5	Shale	JP-5
0.5 (IBP) 1 5 10 20 30 40 50 60 70 80 90 95 99 99.5 (FEP)	112 126 162 174 190 201 211 222 231 242 252 265 273 291 299	234 259 324 345 374 394 412 448 468 509 523 556 570	141 147 164 173 190 202 215 225 235 248 258 273 285 311 323	286 297 327 343 374 396 419 437 455 478 496 523 545 545 613

TABLE 127. HYDROCARBON-TYPE ANALYSIS OF TEST FUELS

	v	olume P	ercent	
Compound Type	Conventional JP-4	Shale JP-4	Conventional JP-5	Shale JP-5
Paraffins Cycloparaffins Dicycloparaffins Alkylbenzenes Indans and tetralins Naphthalenes	67.6 19.8 3.2 7.6 1.8 <0.1	67.9 19.5 1.7 9.6 1.3	47.2 37.7 2.2 7.6 2.9 2.3	62.0 16.9 0.6 8.9 9.0 2.6

2. SIMULATED DISTILLATION OF DF-2 FUEL

A simulated distillation was conducted on a fuel coded 78-8-TJ DF-2. The gas chromatographic method described in ASTM D 2887 was used. Data are presented in Table 128 and a gas chromatogram of the fuel is shown in Figure 73.

TABLE 128. SIMULATED DISTILLATION OF DF-2 FUEL 78-8-TJ DF-2

Percent	Tempe:	rature
Recovered	°C	°F
0.5 (IBP)	95	203
5.0	175	347
10.0	194	381
20.0	216	421
30.0	232	450
40.0	248	478
50.0	260	500
60.0	274	525
76.0	290	554
80.0	308	586
90.0	332	629
95.0	349	659
99.5 (FBP)	390	734

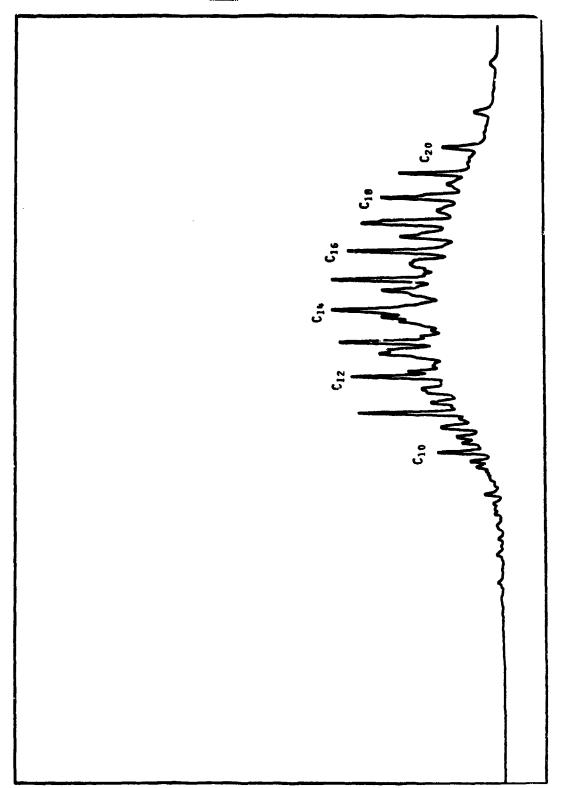


Figure 73. Gas chromatogram of 78-8-TJ DF-2.

3. DENSITY OF DIESEL FUELS

Density as a function of temperature was determined for four diesel fuel specimens by the dilatometer method. The results are shown in Table 129.

TABLE 129. DENSITY OF DIESEL FUELS

	Density, g/cc		C
Sample	32°F	70°F	100°F
78-17-TJ DF-2	0.8481	0.8337	0.8221
78-18-TJ DF-2	0.8490	0.8339	0.8220
78-19-TJ DF-2	0.8485	0.8335	0.8215
78-20-TJ DF-2	0.8489	0.8338	0.8215

4. PROPERTIES AND ANALYSIS OF AROMATIC STOCK (XYLENE BOTTOMS)

An aromatic stock (xylene bottoms) used in Air Force studies for modifying the aromatic content of experimental fuels was characterized by a series of test procedures described in the Appendix. The material was originally thought to consist essentially of xylene isomers, but subsequent analyses showed it to consist mainly of C₃ substituted benzenes.

Kinematic viscosity, density, vapor pressure, and surface tension values as a function of temperature are presented in Table 130. Heat of combustion values are presented in Table 131 and compositional analyses in Table 132. The viscosity/temperature relationship is graphed in Figure 74. The GC analytical trace for the compositional analysis, presented in Figure 75, shows that the sample contained only a small amount of the reported xylene isomers. Further analyses by gas chromatography-mass spectrometry showed that C3 substituted benzenes made up the bulk of the stock.

TABLE 130. PHYSICAL PROPERTIES OF XYLENE BOTTOMS

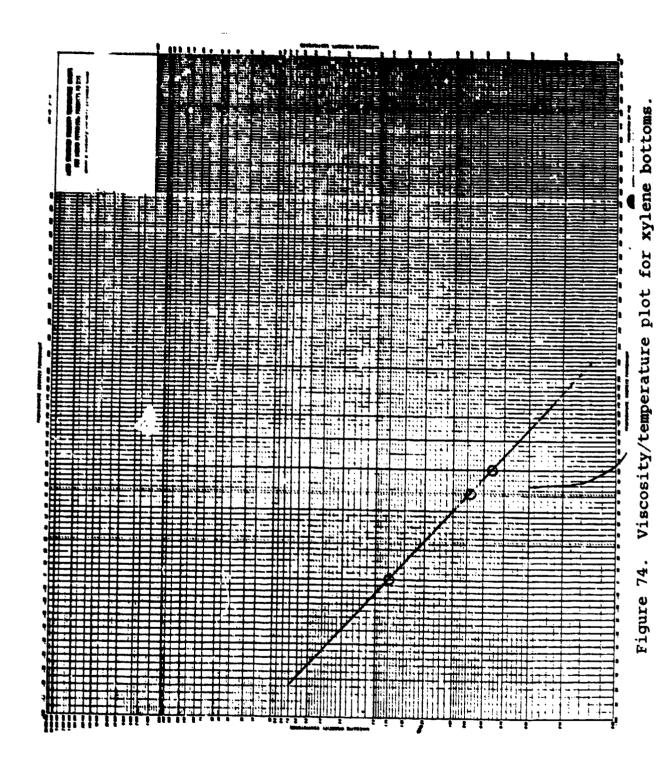
	Temperature,	
Kinematic viscosity, centistokes	0	1.730
	77	0.8784
	100	0.7565
Density, g/cc	32	0.8830
	70	0.8660
	100	0.8524
Vapor pressure, mm Hg	32	3.7
•	70	9.6
	100	18.7
Surface tension, dynes/cm	32	30.56
• • •	70	28.42
	100	26.76

TABLE 131. HEAT OF COMBUSTION OF XYLENE BOTTOMS

Gross,	Btu/lb	Net, Btu/lb
Duplicate	Average	Average
18,594		
18,560		
	18,577	17,710

TABLE 132. GAS CHROMATOGRAPHIC-MASS SPECTROMETRIC ANALYSIS OF XYLENE BOTTOMS

Retention Time, minutes	Volume <u>Percent</u>	Retention Time, minutes	Volume Percent
6.44	0.60	8.01	10.37
6.65	3.79	9.76	8.74
7.16	3.79	9.01	25.24
Total xylenes	8.18	9.05	7.87
_		9.19	8.20
		9.40	7.17
		9.82	19.33
		10.19	0.36
		10.25	0.42
		10.45	1.92
		Total C3 alkylbenze	
		10.55	0.43
		10.69	0.53
		11.30	0.57
		11.40	0.36
		11.48	0.31
		Total C, alkylbenze	



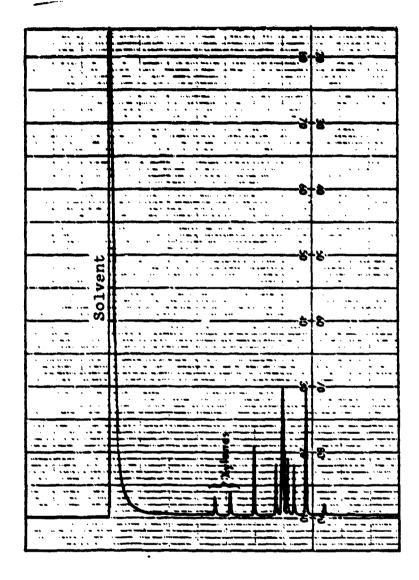


Figure 75. Chromatogram of xylene bottoms.

5. PROPERTIES AND ANALYSIS OF JP-8, TANK F-3

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A multitude of tests were performed on a reference sample of JP-8 fuel in support of an Air Force-sponsored program in combustion technology. The test procedures used are described in the Appendix.

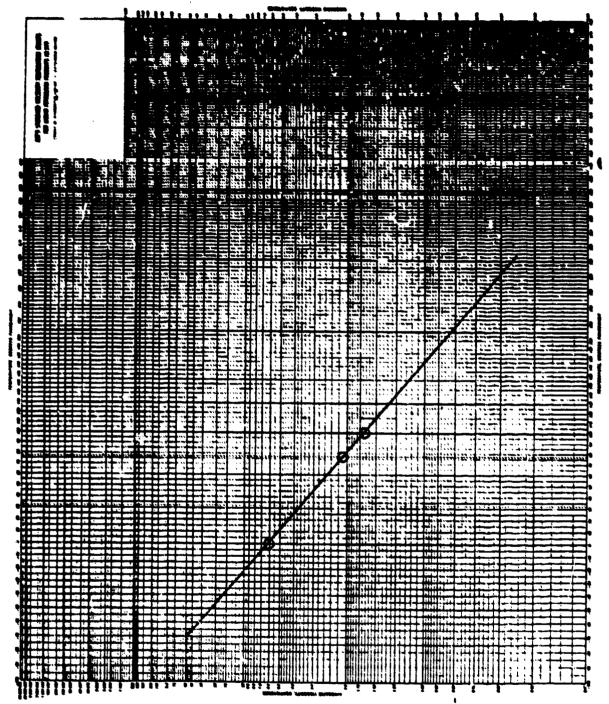
Test results for density, kinematic viscosity, surface tension, and vapor pressure as a function of temperature are shown in Table 133. Heat of combustion values are shown in Table 134, and hydrocarbon type analysis results are presented in Table 135. The temperature/viscosity relationship is illustrated in Figure 76.

TABLE 133. PHYSICAL PROPERTIES OF JP-8, TANK F-3

	Temperature, °F	
Density, g/cc	32	0.8255
	70	0.8102
	100	0.7980
Kinematic viscosity,	0	6.082
centistokes	77	2.080
CCIICIB CONCB	100	1.672
Surface tension,	32	28.81
dynes/cm	70	26.77
1.3 	100	25.14
Vapor pressure,	32	8.4
mm Hg	70	12.7
	100	16.7

TABLE 134. HEAT OF COMBUSTION OF JP-8, TANK F-3

	Btu/lb	Net, Btu/lb
Duplicate	Average	Average
19,786 19,806	19,796	18,519



Viscosity/temperature plot for JP-8, tank F-3. Figure 76.

TABLE 135. HYDROCARBON TYPE ANALYSIS OF JP-8, TANK F-3, BY ASTM D 2789 (MODIFIED)

Compound type	Volume percent		
Paraffins	43.4		
Cycloparaffins	39.8		
Cycloparaffins Dicycloparaffins	3.3		
Alkylbenzenes	7.4		
Indans and tetraling	4.1		
Naphthalenes	2.0		

5. HEAT OF COMBUSTION OF GULF MINERAL SEAL OIL

Gulf Mineral Seal Oil, used for blending special fuels to be used in full-scale combustion tests, was evaluated for net heat of combustion using the oxygen bomb calorimeter according to ASTM procedure D 240-64. The results are given below in Table 136.

TABLE 136. HEAT OF COMBUSTION OF MINERAL SEAL OIL

Gross, I	Stu/lb	Net, Btu/lb		
Duplicate	Average	Average		
19,937				
19,902				
	19,920	18,627		

7. HEAT OF COMBUSTION OF JP-4

JP-4 fuel samples were routinely given in-house full-scale engine tests by the AFAPL Turbine Engine Division. Heat of combustion data were required for these fuels to evaluate their performance in the tests. These data are shown in Table 137.

8. CHEMICAL AND PHYSICAL PROPERTIES OF MODIFIED JP-4 AND JP-8 FUELS

A large number of modified JP-4 and JP-8 fuels were subjected to multiple physical and chemical tests, as described in the Appendix. The number and letter designations after the second

TABLE 137. HEAT OF COMBUSTION OF JP-4 TEST FUELS

		heat of combustion		
	Report	Gross, B	tu/lb	Net, Btu/lb
JP-4 sample	date	Duplicate	Average	Average
Somen tank IDI have	2-7-79	20 127		
Scrap tank, 'D' bay	2-1-13	20,137		
		20,134		
			20,136	18,811
B-3 tank	2-7-79	20,140		
	- , ,,	20,097		
		20,037	20,119	18,794
			20,223	20,734
'D' bay engine cell	2-7-79	20,094		
		20,147		
		,	20,121	18,796
				207.00
B-2 tank	2-7-79	20,076		
		20,107		
			20,092	18,767
Test fuel from	8-31-78	19,934		
AFAPL Turbine		20,008		
Engine Division			19,996	18,686
Test fuel from	10-9-78	20,152		
TF-41 engine	10-3-70	20,111		
test facility		50, 111	20,132	18,818
Cuat Incitica			20,132	10,010
Test fuel from	12-15-78	20,137		
AFAPL full-		20,171		
scale engine		40,41	20,154	18,831
			, -	,
Test facility	3-26-79	20,117		
AFAPL turbine		20,100		
engine test fuel		- •	20,109	18,786
-			•	. • . = =

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hyphen in the sample number indicate the type of fuel (JP-4 or JP-8) and the blending stock used to modify the fuel. The letters AR (2040 aromatic solvent), XY (xylene bottoms), DF-2 (liesel fuel), XG (xylene bottoms and Gulf Mineral Seal Oil) are used. The number following the third hyphen indicates the nominal hydrogen content resulting from the modification (12.0%, 13.0%, or 14.0%).

The data are presented in Tables 138 through 144 and Figures 77 through 108 as follows:

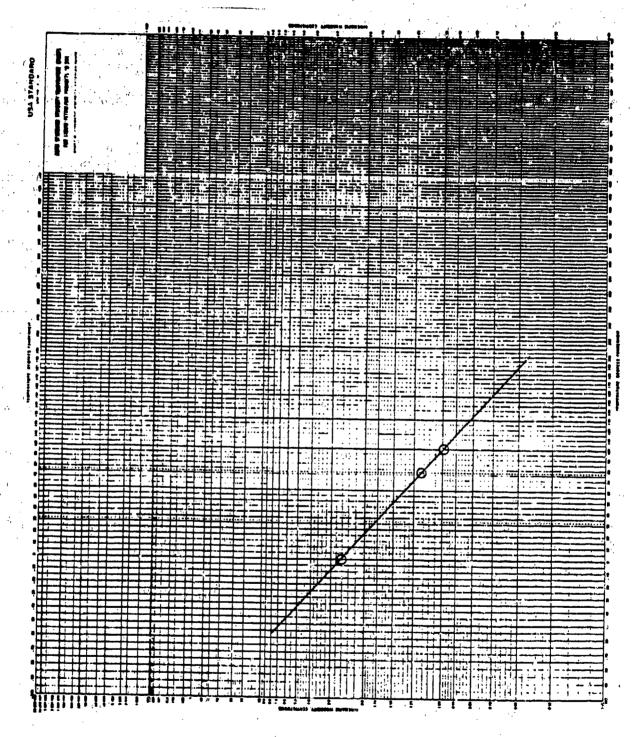
TABLE 138. DENSITY OF MODIFIED FUELS AS A FUNCTION OF TEMPERATURE

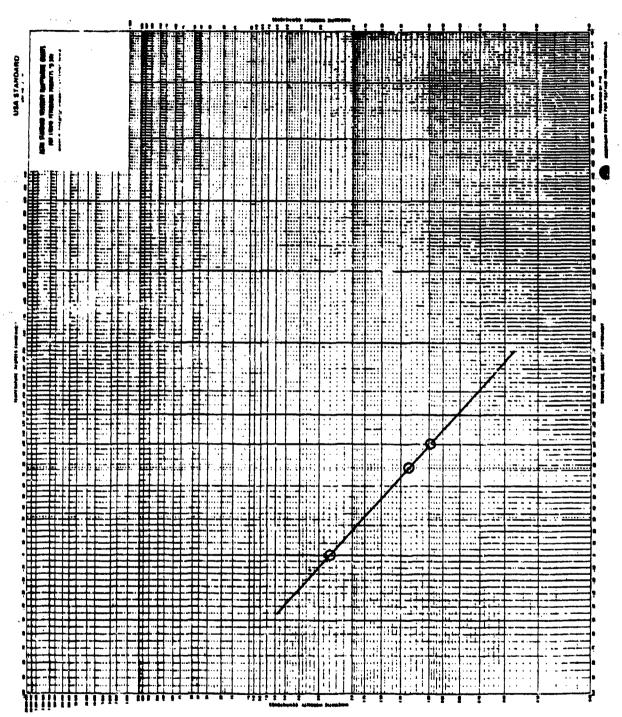
	Density, g/cc		
Sample number	32°F	70°F	100°F
GE/IJ-78-4AR-12.0	0.8506	0.8343	0.8210
GE/TJ-78-4AR-12.0-02	0.8543	0.8375	0.8243
GE/TJ-78-4AR-12.0-03	0.8525	0.8357	0.8225
GE/TJ-78-4AR-12.0-05	0.8489	0.8323	0.8193
GE/TJ-78-4AR-13.0	0.8171	0.3007	0.7878
GE/TJ-78-4AR-13.0-02	0.8168	0.8003	0.7872
GE/TJ-78-4AR-13.0-03	0.8178	0.8010	0.7878
GE/TJ-78-4AR-13.0-05	0.8184	0.8023	0.7895
GE/TJ-78-4XY-12.0 (5/23/78)	0.8305	0.8135	0.8000
GE/TJ-78-4XY-12.0 (5/30/78)	0.8307	0.8134	0.8002
GE/TJ-78-4XY-12.0 (Batch 2)	0.8298	0.8126	0.7990
GE/TJ-78-4XY-12.0-02	0.8306	0.8135	0.7999
GE/TJ-78-4XY-13.0 (5/13/78)	0.8064	0.7899	0.7740
GE/TJ-78-4XY-13.0 (5/30/78)	0.8079	0.7909	0.7771
GE/TJ-78-4XY-13.0 (Batch 2)	0.8081	0.7912	0.7776
GE/TJ-78-4XY-13.0-02	0.8091	0.7922	0.7787
GE/TJ-78-4XG-14.0 (5/23/78)	0.7904	0.7740	0.7608
GE/TJ-78-4XG-14.0 (5/30/78)	0.7908	0.7740	0.7611
GE/TJ-78-4XG-14.0-02	0.7915	0.7749	0.7617
DF-2 (5/25/78)	0.8561	0.8413	0.8295
GE/TJ-78-DF2-13.0-02	0.3491	0.8333	0.8218
GE/TJ-78-8AR-13.0	0.8452	0.8295	0.8171
GE/TJ-78-8AR-13.0-02	0.8454	0.8298	0.8174
GE/TJ-78-8AR-13.0-03	0.8457	0.8300	0.8175
GE/TJ-78-9AR-13.0-05	0.8454	0.8298	0.8177
GE/TJ-78-8AR-12.0-08	0.8728	0.8568	0.8444
GE/TJ-78-8AR-12.0-09	0.8726	0.8568	0.8444
GE/1J-78-8XY-12.0-05	0.8490	0.8326	0.8195
GE/TJ-78-8XY-12.0-08	0.8489	0.8323	0.8191
GE/TJ-78-8XY-12.0-09	0.8486	0.8320	0.8190
GE/TJ-78-8XY-13.0-08	0.8339	0.8180	0.8055
GE/TJ-78-8XY-13.0-09	0.8343	0.8182	0.8053

TABLE 139. KINEMATIC VISCOSITY OF MODIFIED FUELS AS A FUNCTION OF TEMPERATURE

	Viscosi	Viscosity, centistokes		
Sample Number	0°F	77°F	100°F	
GE/TJ-78-4AR-12.0	2.605	1.175	0.989	
GE/TJ-78-4AR-12.0-02	2.598	1.173	0.990	
GE/TJ-78-4AR-12.0-03	2.632	1.173	0.994	
GE/TJ-78-4AR-12.0-05	2.598	1.173	0.988	
GE/TJ-78-4AR-13.0	2.204	1.053	0.903	
GE/TJ-78-4AR-13.0-02	2.199	1.059	0.903	
GE/TJ-78-4AR-13.0-03	2.227	1.063	0.908	
GE/TJ-78-4AR-12.0-05	2.288	1.081	0.920	
GE/TJ-78-4XY-12.0 (5/23/78)	1.623	0.851	0.739	
GE/TJ-78-4XY-12.0 (5/30/78)	1.618	0.851	0.738	
GE/TJ-78-4XY-12.0 (Batch 2)	1.616	0.852	0.738	
GE/TJ-78-4XY-12.0-02	1.662	0.836	0.726	
GE/TJ-78-4XY-13.0 (5/23/78)	1.685	0.864	0.748	
GE/TJ-78-4XY-13.0 (5/30/78)	1.676	0.859	0.745	
GE/TJ-78-4XY-13.0 (Batch 2)	1.662	0.855	0.742	
GE/TJ-78-4XY-13.0-02	1.688	0.864	0.747	
GE/TJ-78-4XG-14.0 (5/23/78)	2.231	1.089	0.927	
GE/TJ-78-4XG-14.0 (5/23/78)	2.236	1.085	0.927	
GE/TJ-78-4XG-14.0-02	2.261	1.093	0.932	
DF-2 (5/25/78)	14.99*	3.356	2.561	
GE/TJ-78-DF2-13.0-02	11.19	3.006	2.318	
GE/TJ-78-8AR-13.0	5.209	1.864	1.508	
GE/TJ-78-8AR-13.0-02	5.212	1.871	1.513	
GE/TJ-78-8AR-13.0-03	5.193	1.861	1.505	
GE/TJ-78-8AR-13.0-05	5.183	1.858	1.511	
GE/TJ-78-8AR-12.0-08	5.409	1.876	1.514	
GE/TJ-78-8AR-12.0-09	5.386	1.870	1.509	
GE/TJ-78-8XY-12.0-05	2.635	1.195	1.007	
GE/TJ-78-8XY-12.0-08	2.633	1.192	1.008	
GE/TJ-78-8XY-12.0-09	2.618	1.196	1.006	
GE/TJ-78-8XY-13.0-08	3.520	1.471	1.223	
GE/TJ-78-8XY-13.0-09	3,569	1.47ú	1.220	

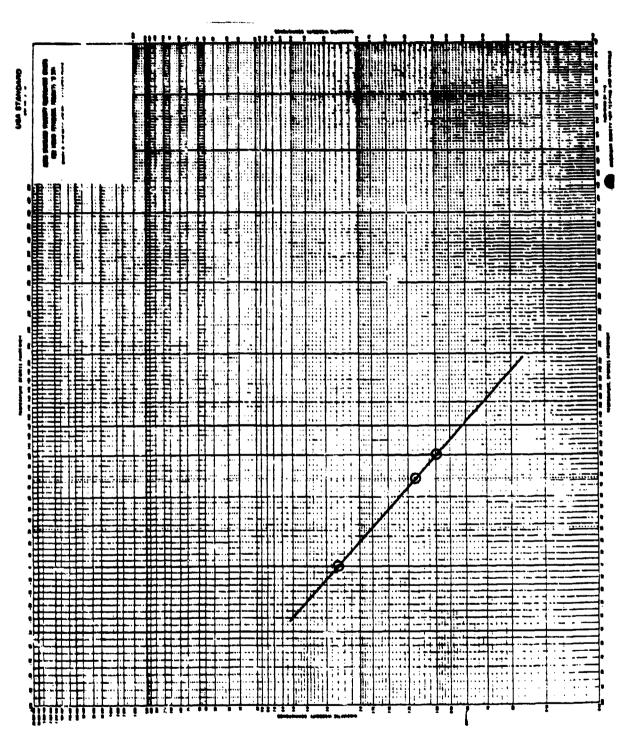
^{*}Forms gel at this temperature.





Viscosity/temperature plot for GE/TJ-78-4AR-12.0-02 Figure 78.

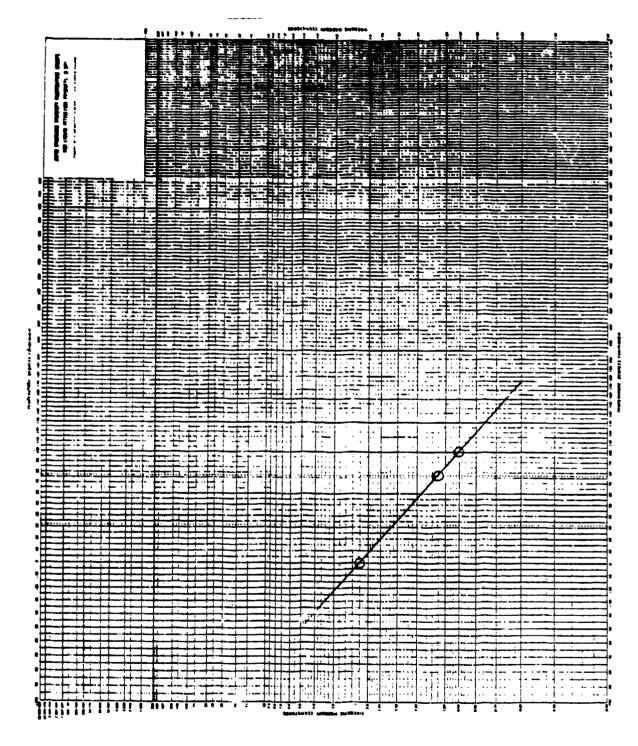
Viscosity/temperature plot for GE/TJ-78-4AR-12.0-03.

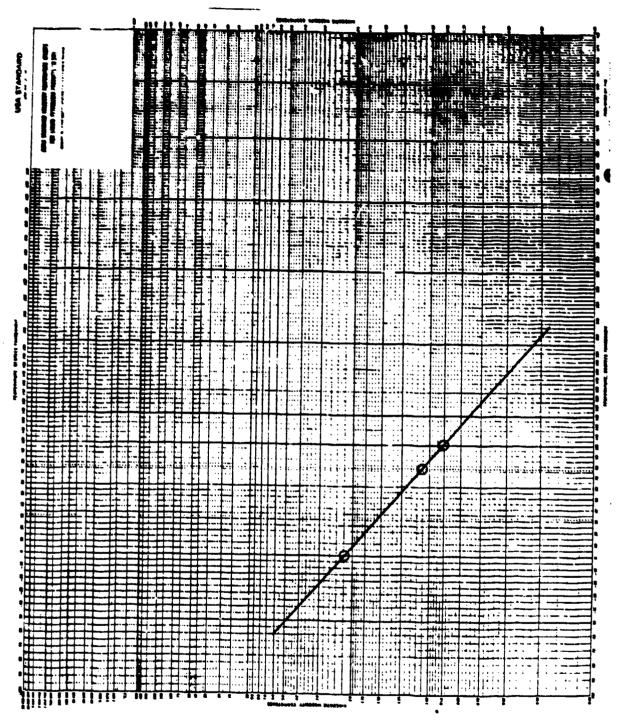


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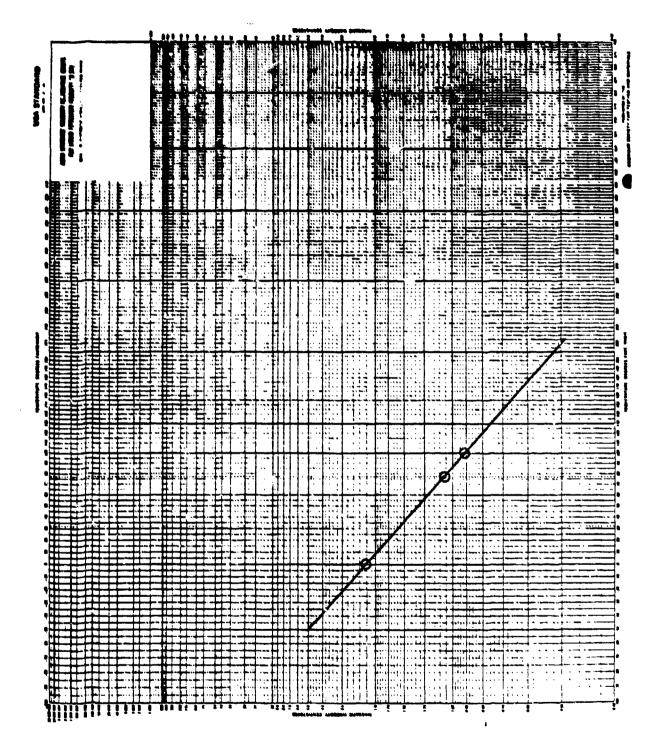
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Viscosity/temperature plot for GE/TJ-78-4AR-12.0-05. Figure 80.

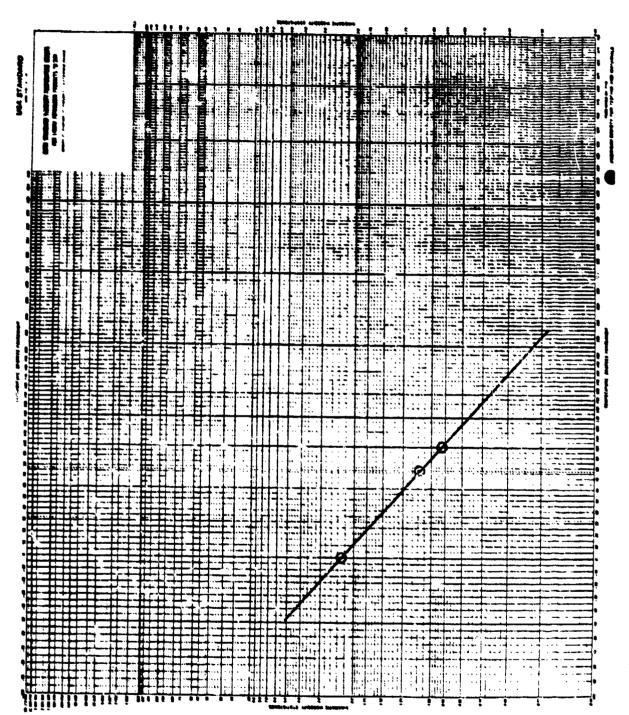




Viscosity/temperature plot for GE/TJ-78-4AR-13.0-02. Figure 82.

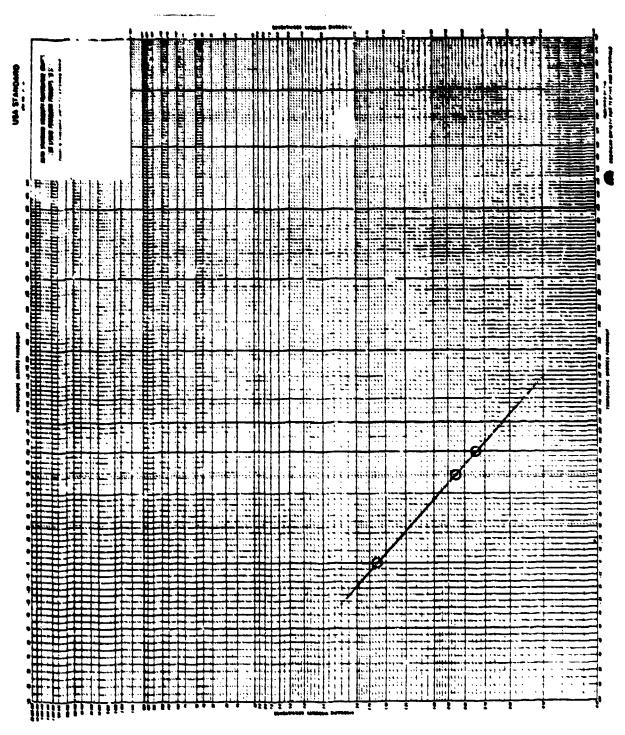


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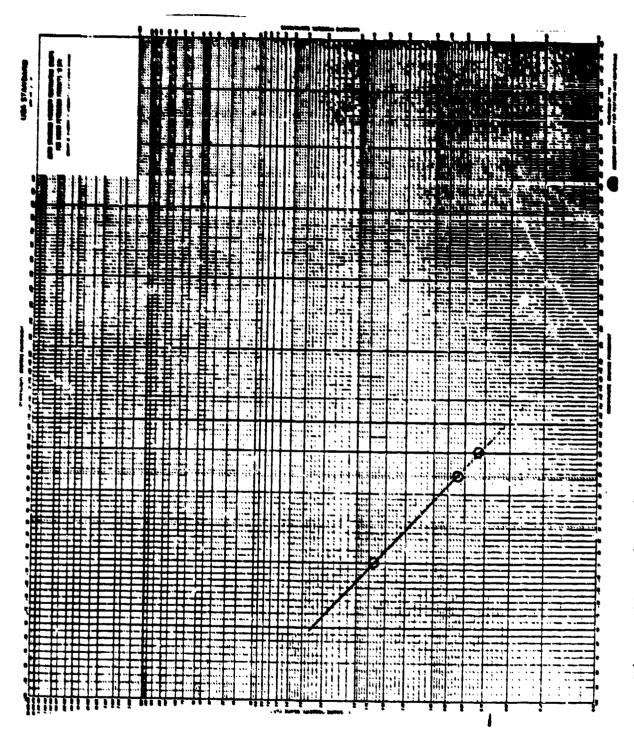
Viscosity/temperature plot for GE/TJ-78-4AR-13.0-05. Figure 84.

Viscosity/temperature plot for GE/TJ-78-4XY-12.0 (5-23-78) Figure 85.

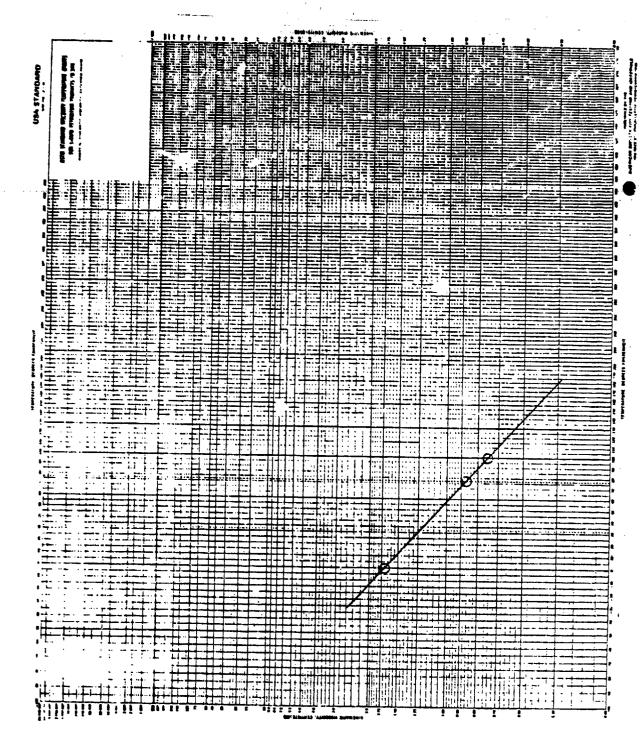


Viscosity/temperature plot for GE/TJ-78-4XY-12.0 (5-30-78) Figure 86.

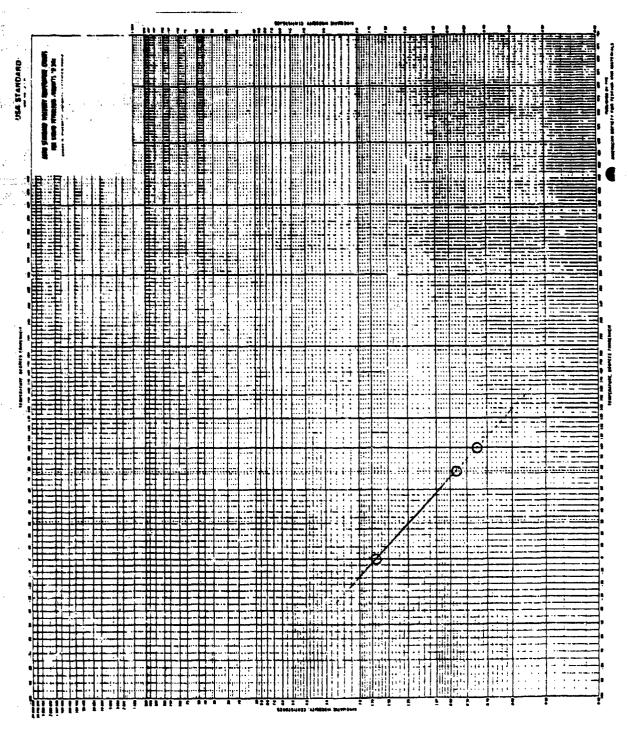
Viscosity/temperature plot for GE/TJ-78-4XY-12.0 (Batch 2) Figure 87.



Viscosity/temperature plot for GE/TJ-78-4XY-12.0-02. Figure 88.

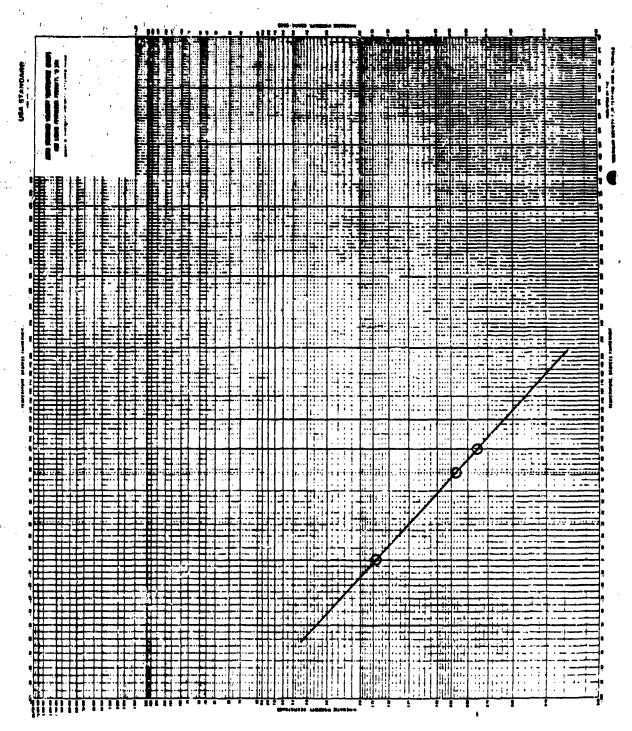


Viscosity/temperature plot for GE/TJ-78-4XY-13.0 (5-23-78)



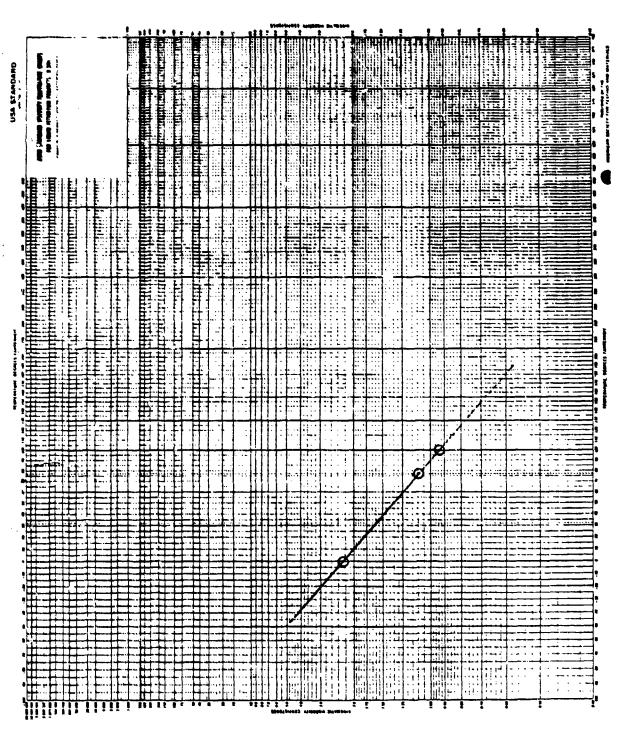
Viscosity/temperature plot for GE/TJ-78-4XY-13.0 (5-30-78) Figure 90.

Viscosity/temperature plot for GE/TJ-78-4XY-13.0 (Batch 2) Figure 91.



Viscosity/temperature plot for GE/TJ-78-4XY-13.0-02

Viscosity/temperature plot for GE/TJ-78-4XG-14.0 (5-23-78) Figure 93.



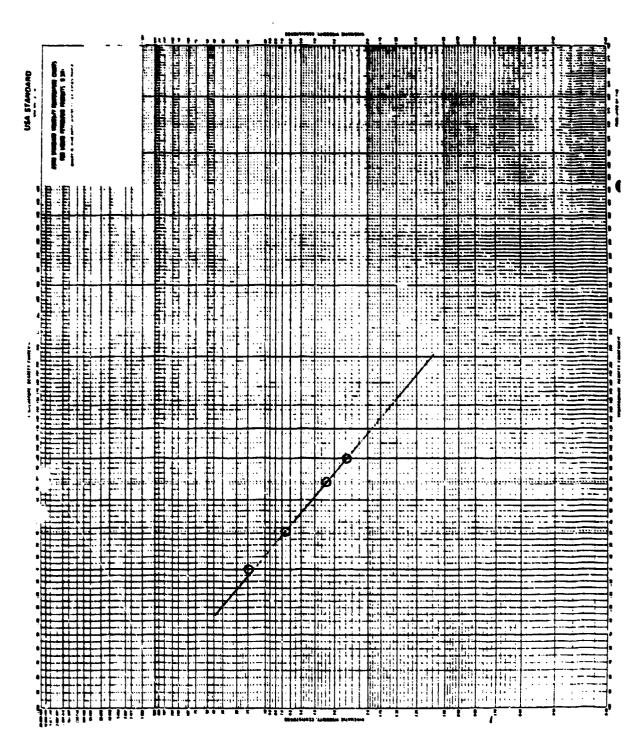
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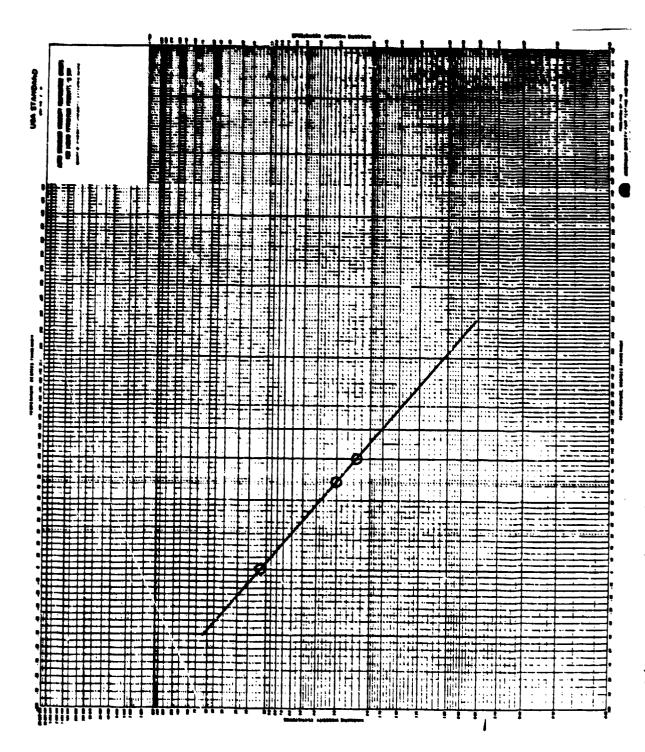
Viscosity/temperature plot for GE/IJ-78-4XG-14.0 (5-30-78)

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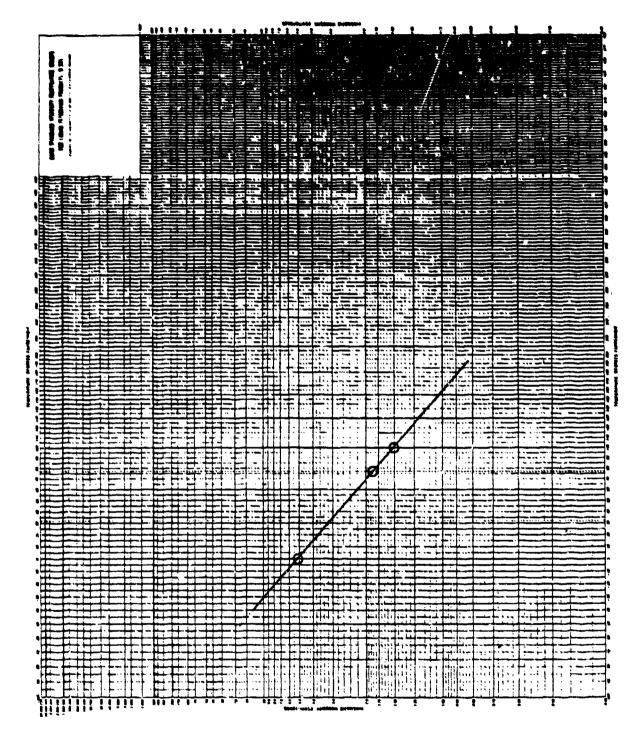
Viscosity/temperature plot for GE/TJ-78-4XG-14.0-02. Figure 95.



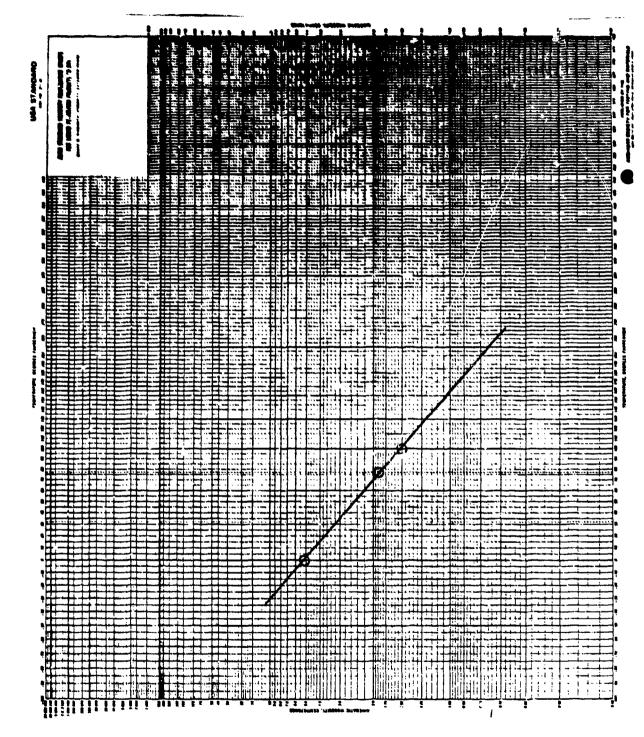
Viscosity/temperature plot for DF-2 (5-25-78). Figure 96.



Viscosity/temperature plot for GE/TJ-78-DF2-13.0-62. Figure 97.



Viscosity/temperature plot for GE/TJ-78-8AR-13.0 Figure 98.



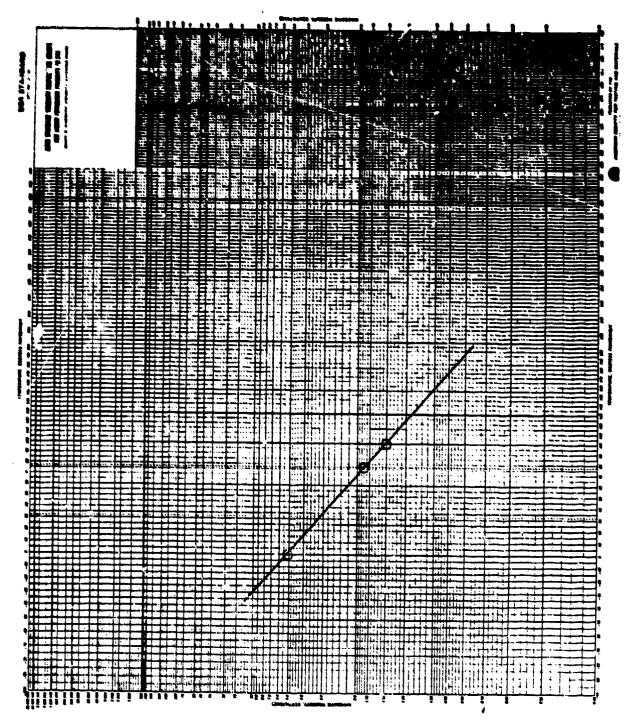


Figure 100. Viscosity/temperature plot for GE/TJ-78-8AR-13.6-03.

Viscosity/temperature plot for GE/TJ-78-8LA-13.0-05. Figure 101.

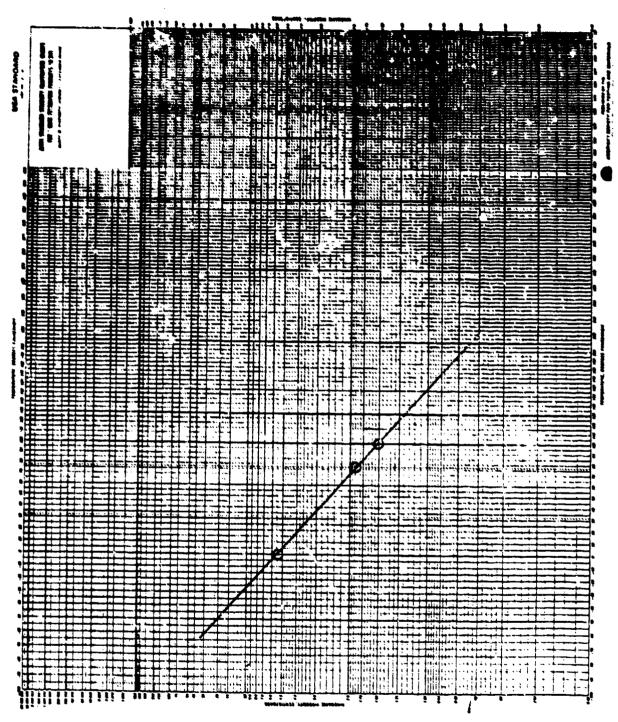


Figure 102. Viscosity/temperature plot for GE/TJ-78-SAR-12.0-08.

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Viscosity/temperature plot for GE/TJ-78-8AR-12.0-09. Figure 103.

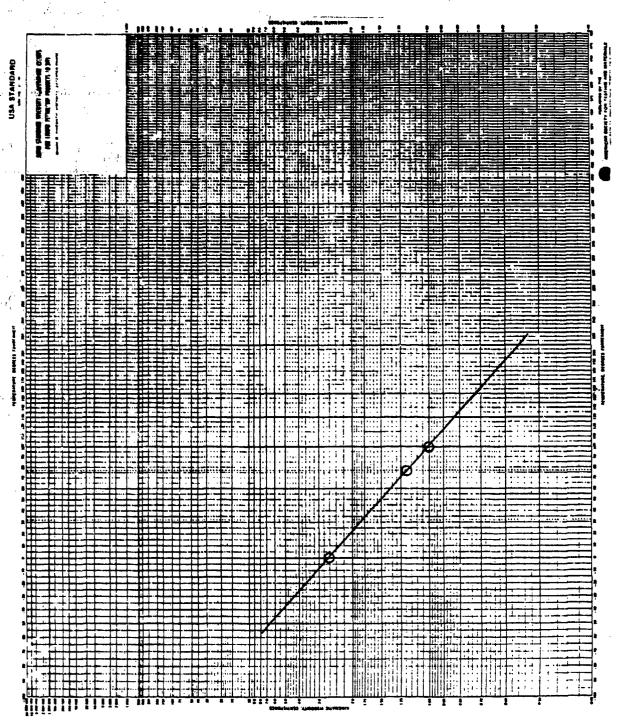
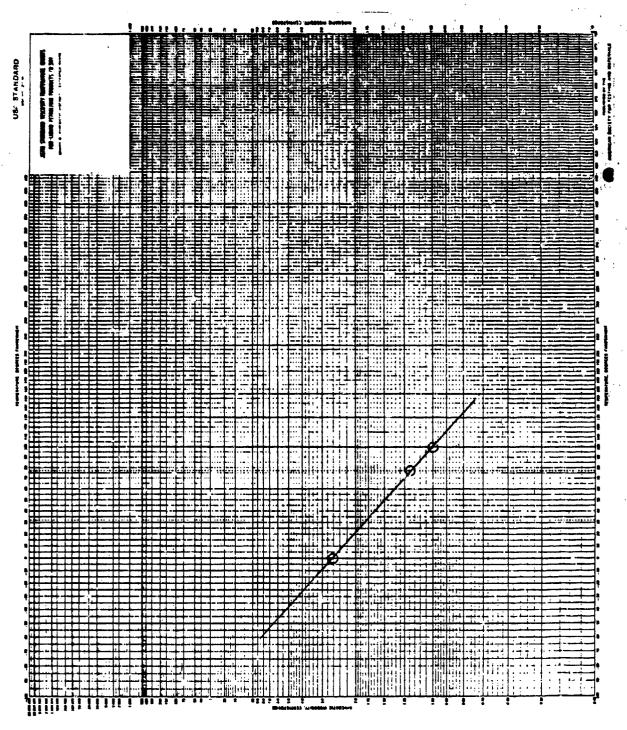


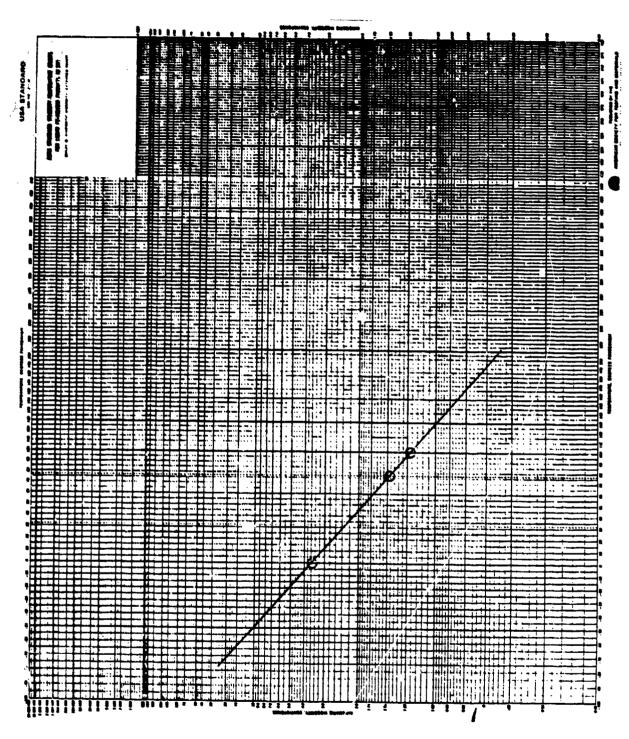
Figure 104. Viscosity/temperature plot for GE/TJ-78-8XY-12.0-05.

Figure 105. Viscosity/temperature plot for GE/TJ-78-8XY-12.0-08



Viscosity/temperature plot for GE/TJ-78-8XY-12.0-09. Figure 106.

Viscosity/temperature plot for GE/TJ-78-8XY-13.0-08.



Viscosity/temperature plot for GE/TJ-78-8XY-13.0-09 Figure 108.

TABLE 140. SURFACE TENSION OF MODIFIED FUELS AS A FUNCTION OF TEMPERATURE

Sample Number	Surface 32°F	Tension, d	ynes/cm
GE/TJ-78-4AR-12.0		-	
·	27.18	25.63	24.42
GE/TJ-78-4AR-12.0-02	27.74	25.83	24.50
GE/TJ-78-4AR-12.0-03	27.56	25.82	24.51
GE/TJ-78-4AR-12.0-05	27.19	25.54	24.41
GE/TJ-78-4AR-13.0	25.62	24.14	23.00
GE/TJ-78-4AR-13.0-02	26.46	24.80	23.45
GE/TJ-78-4AR-13.0-03	26.13	24.58	23.47
GE/TJ-78-4AR-13.0-05	25.91	24.64	23.34
GE/TJ-78-4XY-12.0 (5/23/78)	26.48	25.54	24.31
GE/TJ-78-4XY-12.0 (5/30/78)	27.23	25.60	24.30
GE/TJ-78-4XY-12.0 (Batch 2)	27.34	25.67	24.33
GE/TJ-78-4XY-12.0-02	27.78	25.87	24.36
GE/TJ-78-4XY-13.0 (5/23/78)	25.83	24.36	23.37
GE/TJ-78-4XY-13.0 (5/30/78)	25.67	24.50	23.57
GE/TJ-78-4XY-13.0 (Batch 2)	25.65	24.52	23.61
GE/TJ-78-4XY-13.0-02	25.97	24.48	23.20
GE/TJ-78-4XG-14.0 (5/23/78)	25.45	23.87	22.63
GE/TJ-78-4XG-14.0 (5/30/78	25.38	23.87	22.66
GE/TJ-78-4XG-14.0-02	25.70	24.12	22.97
DF-2 (5/25/78)	29.07	27.73	26.67
GE/TJ-78-DF2-13.0-02	28.53	27.33	26.44
GE/TJ-78-8AR-13.0	28.08	26.77	25.74
GE/TJ-78-8AR-13.0-02	29.78	27.69	26.19
GE/TJ-78-8AR-13.0-03	30.11	27.82	26.36
GE/TJ-78-8AR-13.0-05	29.63	27.53	26.02
GE/TJ-78-8AR-12.0-08	30.42	28.66	27.23
GE/TJ-78-8AR-12.0-09	30.48	28.19	26.36
GE/TJ-78-8XY-12.0-05	29.72	27.47	25.63
GE/TJ-78-8XY-12.0-08	29.40	27.40	25.80
GE/TJ-78-8XY-12.0-09	29.86	27.75	26.05
GE/TJ-78-8XY-13.0-08	29.01	27.17	25.86
GE/TJ-78-8XY-13.0-09	29.02	26.87	25.49

TABLE 141. VAPOR PRESSURE OF MODIFIED FUELS AS A FUNCTION OF TEMPERATURE

		Pressure,	mm Ha
Sample Number	32°F	70°F	100 F
GE/TJ-78-4AR-12.0	18.5	45.0	83.0
GE/TJ-78-4AR-12.0-02	22.5	51.5	93.0
GE/TJ-79-4AR-12.0-03	27.5	60.5	104.0
GE/TJ-78-4AR-12.0-05	23.5	53.5	95.5
GE/TJ-78-4AR-13.0	22.0	53.0	96.0
GE/TJ-78-4AR-13.0-02	28.0	63.0	110.0
GE/TJ-78-4AR-13.0-03	22.5	51.5	94.5
GE/TJ-78-4AR-13.0-05	26.5	60.0	106.0
GE/TJ-78-4XY-12.0 (5/23/78)	19.0	42.0	73.5
GE/TJ-78-4XY-12.0 (5/30/78)	16.5	39.0	72.0
GE/TJ-78-4XY-12.0 (Batch 2)	16.0	37.5	69.0
GE/TJ-78-4XY-12.0-02	19.0	43.0	75.5
GE/TJ-78-4XY-13.0 (5/23/78)	23.5	53.0	93.0
GE/TJ-78-4XY-13.0 (5/30/78)	27.0	59.5	103.0
GE/TJ-78-4XY-13.0 (Batch 2)	25.5	55.5	99.0
GE/TJ-78-4XY-13.0-02	25.5	56.5	99.0
GE/TJ-78-4XG-14.0 (5/23/78)	22.5	50.5	89.5
GE/TJ-78-4XG-14.0 (5/30/78)	28.5	63.0	112.0
E/TJ-78-4XG-14.0-02	25.5	56.5	98.0
DF-2 (5/25/78)	6.5	10.5	15.0
GE/TJ-78-DF2-13.0-02	5.0	9.0	14.0
GE/TJ-78-8AR-13.0	5.0	9.0	13.5
GE/TJ-78-8AR-13.0-02	5.0	9.0	13.5
GE/TJ-78-8AR-13.0-03	5.5	10.0	15.0
GE/TJ-78-8AR-13.0-05	6.0	11.5	17.0
GE/TJ-79-8AR-12.0-08	9.0	14.0	19.5
61/T - 8AR-12.0-09	6.0	10.0	14.0
GE/TJ-78-8XY-12.0-05	3.5	8.0	15.5
GE/TJ-78-8XY-12.0-08	3.5	9.0	17.0
GE/TJ-78-8XY-12.0-09	4.0	9.5	18.0
GE/TJ-7 Y-13.0-08	6.0	12.5	21.0
GE/TJ-/%-8XY-13.0-09	6.0	12.5	21.0
GE/TJ-78-8AR-12.0-01	4.2	7.5	11.5
GE/TJ-78-8XY-12.0-01	3.6	8.2	14.5
GE/TJ-78-8XY-13.0-0	4.0	9.1	16.3

TABLE 142. HEAT OF COMBUSTION OF MODIFIED FUELS

	Gro	ss, Btu/	1b	Net, Btu/1b
Sample Number	Dupli	cates	Avg	Avg
GE/TJ-78-4AR-12.0	19,243	19,217	19,230	18,144
GE/TJ-78-4AR-12.0-02	19,204	19,194	19,199	18,111
GE/TJ-78-4AR-12.0-03	19,127	19,148	19,138	18,054
GE/TJ-78-4AR-12.0-05	19,141	19,140	19,141	18,042
GE/TJ-78-4AR-13.0	19,490	19,531	19,511	18,327
GE/TJ-78-4AR-13.0-02	19,486	19,477	19,482	18,291
GE/TJ-78-4AR-13.0-03	19,490	19,483	19,487	18,313
GE/TJ-78-4AR-13.0-05	19,437	19,467	19,452	18,213
GE/TJ-78-4XY-12.0 (5/23/78)	19,207	19,256	19,232	18,141
GE/TJ-78-4XY-12.0 (5/30/78)	19,263	19,268	19,266	18,178
GE/TJ-78-4XY-12.0 (Batch 2)	19,364	19,340	19,352	18,256
GE/TJ-78-4XY-12.0-02	19,223	19,192	19,208	18,117
GE/TJ-78-4XY-13.0 (5/23/78	19,530	19,532	19,531	18,350
GE/TJ-78-4XY-13.0 (5/30/78)	19,533	19,563	19,548	18,369
GE/TJ-78-4XY-13.0 (Batch 2)	19,459	10,408	19,434	18,253
GE/TJ-78-4XY-13.0-02	19,552	19,515	19,534	18,350
GE/TJ-78-4XG-14.0 (5/23/78)	19,936	19,966	19,951	18,674
GE/TJ-78-4XG-14.0 (5/30/78)	19,843	19,787	19,815	18,538
GE/TJ-78-4XG-14.0-02	19,865	19,828	19,847	18,573
DF-2 (5/25/78)	19,567	19,543	19,555	18,354
GE/TJ-73-DF2-13.0-02	19,613	19,596	19,605	18,393
GE/TJ-78-8AR-13.0	19,453	19,507	19,480	18,296
GE/TJ-78-8AR-13.0-02	19,501	19,462	19,482	18,305
GE/TJ-78-8AR-13.0-03	19,488	19,482	19,485	18,305
GE/TJ-78-8AR-13.0-05	19,455	19,492	19,474	18,291
GE/TJ-78-8AR-12.0-08	19,161	19,150	19,156	18,063
GE/TJ-78-8AR-12.0-09	19,144	19,162	19,153	18,059
GE/TJ-78-8XY-12.0-05	19,216	19,200	19,208	18,111
GE/TJ-78-8XY-12.0-08	19,204	19,205	19,205	18,112
GE/TJ-78-8XY-12.0-09	19,171	19,200	19,186	18,085
GE/TJ-78-8XY-13.0-08	19,458	19,512	19,485	18,301
GE/TJ-78-8XY-13.0-09	19,518	19,500	19,509	18,327

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GAS CHROMATOGRAPHIC SIMULATED DISTILLATION OF MODIFIED FUELS TABLE 143.

STATE AND SECTION OF THE PROPERTY OF THE PROPE

Z/IJ-78-	4AR-13.0	89	86	154	87 187	237	271	322	361	397	426	451	487	514	267	581	
GE/T.	-CC	20	30	8	87	114	133	161	193	203	219	233	253	268	297	305	
-78-	•P	9	82	158	190	235	284	338	378	406	439	460	491	522	570	888	
GE/T3	4AR-12.0-05	19	58	70 158	88	113	140	170	192	208	226	238	255	272	299	309	
-78-	• F	2	73	145	185	234	280	333	374	405	439	464	200	525	899	585	
GE/TJ-78-	4AR-12.0-03	18	23	63 145	85	112	138	167	190	202	226	240	360	274	298	307	
-8/	- 102 - 102	2	75	149	184	234	62	131	071	501	137	162	361	122	.67	181	
GE/TJ-	4AR-12.0-02	18 64	24	65	85	112	137	166	188	207	225	239 4	257 4	272	297	305	
78-	0 4	75	16	176	203	246	300	354	388	417	146	1 68	495	522	565	574	
1	∾i'				-		- •	•	•	•	•	•	•			-•	
GE/TJ-78-	4AR-12.0	24	33	80	95	119	149	179	198	214	230	242	257	272	296 565	301	

(continued)

(continued)

TABLE 143 (continued)

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GE/TJ-78- 4XY-13.0 (Batch 2)	1.	ij	3	138	176	219	259	286	316	342	378	417	457	482	527	549
GE/TJ-78 4XY-13.0 (Batch 2	ر ا•	17	19	59	8	10	126	141	158	172	192	214	236	250	275	287
GE/TJ-78- 4XY-13.0 (5/30/78)	1	61	8	138	173	223	261	284	316	340	376	41.5	455	482	531	554
GE/TJ-78 4XY-13.0 (5/30/78	4. 0.	16	19	59	81	106	127	140	158	171	161	213	235	250	772	290
GE/TJ-78- 4XY-13.0 (5/23/78)	•	2	72	144	183	230	3 66	291	320	345	381	421	460	486	525	541
GE/TJ-78 4XX-13.0 (5/23/78	9	18	22	62	84	110	130	144	160	174	194	216	238	252	274	283
-78-	AI.	2	79	153	187	235	275	295	318	337	367	412	453	484	532	267
GE/TJ-78- 4xy-12.0-02	2	18	5 6	67	98	113	135	146	159	169	136	211	234	251	278	297
-78- 2.0 h 2)	a l	9	81	144	181	232	271	288	315	329	354	396	444	475	522	547
GE/TJ-78- 4XY-12.0 (Batch 2)	ပ္	19	27	62	83	111	133	142	157	165	179	202	229	246	272	286
1-78- 2.0 1/78)	•	64	11	151	185	232	271	288	315	333	358	403	448	475	516	538
GE/TJ-78- 4xx-12.0 (5/30/78)	ပ္	18	25	99	85	111	133	142	157	167	181	206	231	246	569	281
GE/TJ-78- 4XY-12.0 (5/23/78)	4	61	75	145	183	232	271	291	316	334	367	408	450	478	520	543
• •		16	24	63	8	111	133	144	158	168	186	209	232	248	271	284
1-78- 1.0-05	4	63	73	145	183	229	266	316	358	394	424	451	486	514	572	265
GE/TJ-78- 4AR-13.0-05	ပ	11	23	63	8	109	130	158	181	201	218	233	252	268	300	314
-78-	d.	19	73	142	180	225	262	307	349	385	417	450	486	511	547	563
GE/TJ-78- 4AR-13.0-03	္င	16	23	19	82	107	128	153	176	196	214	232	252	566	286	295
-78- 0-02	4	19	99	143	183	228	264	311	354	390	421	450	486	513	556	574
GE/TJ-78- 4AR-13.0-02	ပ	16	19		84			155						267	291	301
Percent	Recovered	0.5, IBP	1	ĸ	10	20	30	40	50	09	70	80	06	95	66	99.5, FBP

TABLE 143 (continued)

GE/TJ-78- 8MR-13.0-03	207	246	315	338	367	392	412	435	455	478	203	532	554	286	604
	76	119	157	170	186	200	211	224	2.15	248	361	278	28	308	318
GE/TJ-78- 8AR-13.0-02	241	261	318	340	367	392	414	435	455	480	200	532	226	286	604
GE/TJ-78- 8AR-13.0-0	116	127	159	171	186	200	212	224	235	249	36 2	378	291	308	318
GE/TJ-78- BAR-13.0	244	3 66	315	336	365	387	406	428	448	468	487	511	541	583	909
.	118	130	157	169	185	197	208	220	231	242	253	266	283	306	319
GE/TJ-78- DF2-13.0-02	223	259	329	358	396	424	451	480	507	538	574	613	653	723	745
GE/T. DF2-1	106	126	165	181	202	218	233	249	264	281	301	323	345	384	396
DF-2 (5/25/78)	208	248	334	369	408	441	469	496	523	554	585	622	662	725	762
	86	120	168	187	209	227	243	258	273	290	307	328	350	385	406
GE/TJ-78- 4XG-14.0-02	2	72	144	183	232	275	304	340	381	426	471	549	588	630	646
68/m 6x6-1	18	22	62	8	111	135	151	171	194	219	244	287	309	332	341
6xG-14.0- (5/30/78)	24	99	138	178	226	268	306	345	388	432	478	558	290	640	662
	12	19	29	81	108	131	152	174	198	222	248	292	310	338	350
GE/TJ-78- 4XG-14.0- (5/23/78)	19	8	138	180	228	270	307	343	387	430	475	554	288	630	651
	16	20	59	83	109	132	153	173	197	221	246	290	309	332	344
GE/TJ-78- XY-13.0-02	64	8	142	181	228	3 66	291	318	345	380	421	460	484	520	541
GE/TJ-78- 4XY-13.0-02	18	19	61	83	109	130	144	159	174	194	216	238	251	271	283
Percent Recovered	0.5, IBP		S	10	20	30	•	20	90	70	80	8	95	66	99.5, FBP

(continued)

TABLE 143 (continued)

	ļ	1	•	ļ	•	1		i								
Percent Recovered	60E/T.	GE/TJ-78- 8AR-13.0-05 °C °F	9AR-11	GE/TJ-79- 8AR-12.0-08 •C •F	CE/TJ-78- BAR-12.0-09 C *F	-78-	6XY-12.0-05	-78-	6XY-12.0-08	-86-0.	6XY-12.0-09		CE/TJ-78- 8XY-13.0-08	-78-	622/73-78- 8XY-13.0-09	-96-0.
0.5, TBP	1 21	230	801	226	121	250	8	228	121	250	120		107	225	120	248
	124		125	257	135	275	127	561	122	276	131	268	121	250	129	264
	157	315	162	324	163	325	142	882	142	887	143	289	143	289	123	289
10	170		172	342	173	343	148	396	147	297	148	298	151	305	152	306
	186		189	372	190	374	164	327	163	325	191	322	168	334	991	334
	200		203	397	203	397	173	343	172	342	172	342	182	360	181	358
	211		215	419	215	419	186	367	186	367	186	367	197	387	196	385
	224		227	#1	22.7	441	202	396	202	326	201	394	212	414	210	410
	236		237	459	238	460	218	424	219	426	218	424	226	439	777	435
	249		251	484	251	484	233	451	234	453	233	451	240	79	238	460
	261		264	507	263	505	250	482	250	482	250	482	257	495	255	491
	278		280	536	280	536	270	518	172	520	270	518	275	527	274	\$25
95	167		262	558	292	558	285	545	586	547	285	545	790	554	288	550
	307		900	586	ŝ	336	303	281	306	583	306	583	307	28 2	306	583
99.5, FBP	312		316	601	314	597	312	594	314	597	314	597	312	265	310	290

HYDROCARBON-TYPE DISTRIBUTION OF MODIFIED FUELS TABLE 144.

				Volume Percent	rcent			
Compound Type	GE/TJ-78- 4AR-12.0	GE/TJ-78-	GE/TJ-78- 4AR-12.0-03	CE/E3-78- CNR-12.0-05	GE/T3-78-	GE/T3-78-	CZ/T2-76-	448-13.0-05
Parattins	38.4	37.6	37.6	38.0	47.2	*	45.9	6.0
Monocycloparaffina	20.4	21.2	20.7	21.1	25.4	4.62	25.3	75.4
Dicycloparaffins	ı	•	•	•,	•	•	ı	•
Alkylbenzenes	12.4	12.7	12.7	12.6	10.7	11.0	11.2	11.0
Indens and tetraline	4.4	4.5	4.5	**	3.0	3.1	3.2	3.2
Maphthalenes	24.4	24.0	24.5	23.9	13.7	14.3	14.4	14.4
	GE/TJ-78- 4XY-12.0		• •	GE/13-70-	GE/TJ-78- 4XY-13.0		_	Q/13-78-
	12/23/1	(5/30/78)	(Batch Z)	4XX-12.0-02	(5/23/78)	(81/06/5)	(Batch 2) 4X	4X7-13.0-02
Paraffins	33.3	33.0	33.0	33.1	45.8	45.8	45.2	46.0

33.4 33.4

15.1

15.1 3.4 34.7

15.1 3.4 34.7

11.7

2.11

2.4 53.0

Monocycloparaffins Dicycloparaffins Alkylbenzene

2.7

11.3

52.9

53.1

3.3

35.5

9.5

0.2

Trace 0.3

Trace

Indias and tetralins

Naphthalenes

6.3

6.1

TABLE 144 (continued)

				Volume Percent	ercent			
	GE/TJ-78- 4XG-14.0 (5/23,78)	GE/TJ-78- GE/TJ-78- 4XG-14.0 4XG-14.0 (5/23/78) (5/30/78)	GE/TJ-78- 4xG-14.0-02	DF-2 (5/25/78)	GE/TJ-78- DF2-13.0-02	GE/TJ-78- BAR-13.0	GE/TJ-78- GE/TJ-78- BAR-13.0 BAR-13.0-02	CZ/TJ-78- 8AR-13.0-03
Paraffins	58.4	58.4	57.7	4.9	44.9	39.1	38.3	38.2
Monocycloparaffins	20.1	20.1	20.9	32.7	32.7	34.1	33.9	34.0
Dicycloparaffins	5.0	5.0	5.3	2.5	2.5	1.5	1.7	ž.6
Alkylbenzenes	14.8	14.8	14.1	8.6	9.8	9.6	9.6	9.6
Indans and tetralins	1.1	1.1	1.3	6.1	. 6.1	3.8	4.2	4.2
Naphthalenes	9.0	9.0	0.1	5.2	5.2	12.0	12.3	12.4

	GE/TJ-78- 8AR-13.0-05	GE/TJ-78- 8AR-12.0-08	GE/13-78- 8ar-12.0-09	CE/TJ-78- 8XY-12.0-05	CE/TJ-78- BXY-12.0-08	GE/TJ-78- 8XY-12.0-09	CE/TJ-78- 8XY-13.0-08	GE/TJ-78- 8XY-13.0-09
Paraffins	38.3	31.2	31.2	26.4	26.4	26.4	36.2	36.3
Monocycloparaffins	33.9	27.9	27.6	22.3	22.3	22.1	31.8	31.5
Dicycloparaffins	1.7	1.3	1.5	1.6	1.5	1.6	1.8	1.9
Alkylbenzene3	9.5	11.5	11.5	48.8	48.9	49.0	27.9	28.1
Indans and tetralins	4.2	5.4	5.4	•	•	<u>.</u>	6.0	6.0
Naphthalenes	12.4	22.7	22.8	6.0	6.0	6.0	1.4	1.3

Density by the dilatometer method - Table 138,

Kinematic viscosity by ASTM D 445 - Table 139 and Figures 77-108,

Surface tension by the capillary rise method - Table 140,

Vapor pressure by the micro-method - Table 141,

Heat of combustion by ASTM D 240 - Table 142,

GC simulated distillation by ASTM D 2887 - Table 143, and

Hydrocarbon type analyses by ASTM D 2789 - Table 144.

9. CHEMICAL AND PHYSICAL PROPERTIES OF FUELS TESTED IN TF41 COMBUSTOR AND IN J79 LOW-SMOKE COMBUSTOR

Chemical and physical properties were determined for a large number of experimental fuels and blending components scheduled for testing combustors. The analytical data, which were required for correlation with the combustion performances of the fuels, are presented in Tables 145 through 152 and Figures 109 through 133 as follows:

Density by the dilatometer method - Table 145,
Kinematic viscosity by ASTM D 445 - Table 146 and Figures 109-133,
Surface tension by the capillary rise method - Table 147,
Vapor pressure by the micro-method - Table 148,
Specific gravity and API gravity from density - Table 149,
Heat of combustion by ASTM D 240 - Table 150,
GC simulated distillation by ASTM D 2887 - Table 151,
Hydrocarbon type analysis by ASTM D 2789-71 - Table 152.

Specific gravity and API gravity were calculated from density data determined by the dilatomer method. All other analytical methods are described in the Appendix.

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The sample number coding system for the fuels presented in this subsection differs slightly from the code used for the samples shown in the previous subsection. The fuels with codes terminating in "(2006).78-C" were tested in the TF41 combustor. The

TABLE 145 DENSITY OF TESTED FUELS AS A FUNCTION OF TEMPERATURE

		Density	, g/cc	
Sample Designation	-20°F	32°F	70°F	100°F
JP-8 (2006), 78-C		0.8252	0.8099	0.7977
8A2 " "	•	0.8755	0.8600	0.8480
8A3 ". "	1.	0.8495	0.8343	0.8225
JP-4 (2006), 78-C		0.7739	0.8574	0.8445
4A2 " "		0.8483	0.8322	0.8193
4A3 " "		0.8183	0.8017	0.7887
8X2 " "	•	0.8515	0.8352	0.8222
8X3 " "		0.8389	0.8230	0.8106
8GM " "	•	0.8264	0.8113	0.7994
4X2 " "	. *	0.8324	0.8153	0.8021
4X3 " "	•	0.8083	0.7913	0.7780
4XG " "	1	0.7928	0.7763	0.7636
GEC-120-8X0-792033	0.8702	0.8480	0.8314	0.8188
GEC-130-4X0-792033	0.8282	0.8049	0.7878	0.8848
GEC-130-DF2-792033	· 🕳	0.8589	0.8434	0.8319
GEC-145-400-792033	0.7957	0.7723	0.7557	0.7423
GEC-140-300-792033	0.8460	0.8252	0.8096	0.7977
GEC-130-8X0-792033	0.8598	0.8380	0.8219	0.8097
GEC-140-4GX-792033	0.8100	0.7876	0.7712	0.7580
GEC-120-8A0-792033	0.8967	0.8746	0.8589	0.8469
GEC-130-8AO-792033	0.8706	0.8488	0.8333	0.8213
GEC-120-4AO-792033	0.8709	0.8475	0.8309	0.8182
GEC-130-4A0-792033	0.8393	0.8156	0.7990	0.7858
GEC-120-X40-792033 Feb	0.8519	0.8281	0.8110	0.7980
GEC-140-8GO-792033 Feb	0.8466	0.8255	0.8104	0.7986

TABLE 146. KINEMATIC VISCOSITY OF TESTED FUELS AS A FUNCTION OF TEMPERATURE

•	•		Centis			
Sample Designation	-20°F	0°F	32°F	70°F	77°F	100°F
JP-8 (2006), 78-C		6.068			2.081	1.670
8A2 " "		5.816			1.964	1.576
8A3 " "		5.841			2.004	1.610
JP-4 (2006), 78-C	A) *	1.854		**	0.948	0.819
4A2 " "		2.582			1.143	0.966
4A3 " "		2.208			1.063	0.907
8x2 " . "	**	2.786			1.244	1.047
8X3 " "		3.900			1.562	1.289
8GM " "		7.075			2.316	1.841
4x2 " "		1.705			0.884	0.764
4x3 " "		1.742			0.910	0.785
4XG " ' '		2.342		•	1.126	0.957
GEC-120-8X0-792033	3.418		1.844	1.240		0.988
GEC-130-4X0-792033	2.084		1.219	0.910		0.752
GEC-130-DF2-792033	-		5.774	3.267	3.004	2.306
GEC-145-400-792033	2.206		1.288	0.955	0.911	0.786
GEC-140-800-792033	9.101		3.526	2.233	2.075	1.665
GEC-130-8X0-792033	5.655		2.516	1.688	1.583	1.304
GEC-120-X40-792033			, ,	1.172		0.948
GEC-140-4GX-792033	2.753		1.531	1.113		0.903
GEC-140-8GO-792033				1.862		1.423
GEC-120-8A0-792033	9.053		3.391	2.134		1.591
GEC-130-8AO-792033	9.020		3.431	2.169		1.620
GEC-120-4AO-792033	3.304		1.688	1.206		0.963
GEC-130-4A0-792033	2.671		1.461	1.072	*	0.871
GEC-120-X40-792033 Feb	2.077		1.202	0.900		9.744
GEC-140-8GO-792033 Feb	10.70		3.938	2.451		1.807

TABLE 147. SURFACE TENSION OF TESTED FUELS AS A FUNCTION OF TEMPERATURE

					Dynes per	centimeter	
San	ple des	ignatio	on	-20°Fª	32°F	70°F	100°F
JP-8	(2006),	78-C			27.56	23.81	24.44
8A2	11	11			29.33	27.47	25.99
8A3	H	il			28.59	26.83	25.43
JP-4	(2006),	78-C			24.67	22.65	21.05
4A2	11	11			26.50	24.78	23.45
4A3	11	u			25.85	23.94	22.50
8X2	11	17			30.00	27.95	26.33
8X3	11	11			29.59	27.45	25.79
8GM	11	ti.			29.10	27.13	25.58
4X2	11	17			27.90	25 .95 .	24.43
4X3	11	11			26.62	24.49	22.84
4XG	11	11			26.10	24.18	22.73
GEC-1	20-8X0-	792033		32.80 ^a	29.85	27.82	26.20
GEC-1	L30-4X0-	792033		28.60	26.35	24.73	23.43
GEC-1	L30-DF2-	792033		32.10	30.07	28.60	26.68
GEC-	L45-400-	792033		27.52	25.05	23.28	21.73
GEC-	L40-800-	792033		31.17	28.78	27.08	25.69
GEC-	L30-8X0-	792033		31.80	29.47	27.80	26.46
GEC-	L40-4GX-	792033		28.21	25.90	24.20	22.86
GEC-	L20-8AO-	792033		33.00	29.98	27.77	25.05
GEC-	L30-8A0-	792033		31.83	29.03	27.00	25. 30
GEC-	L20 -4A 0-	792033		29.24	26.55	24.59	23.03
GEC-	L30-4A0-	792033		28.08	25.50	23.62	22.10
GEC-	L20-X40-	792033	Feb	29.28	26.70	24.81	23.30
GEC-	140-8GO-	792033	Feb	31.08	28.47	26.55	25.05

aResults at this temperature were extrapolated from higher temperature data.

TABLE 148. VAPOR PRESSURE OF TESTED FUELS AS A FUNCTION OF TEMPERATURE

		Milli	meters, me	ercury
Sample designation	on	32°F	70°F	100°F
JP-8 (2006), 78-C		5.0	8.0	11.0
8A2 " "		8.5	13.0	17.0
8A3 " "		8.0	12.5	16.5
JP-4 (2006), 78-C		46.5	95.0	160.0
4A2 " "		24.0	52.5	91.5
4A3 " "		23.0	53.5	98.0
8X2 " "		7.0	13.5	21.5
8X3 " "		10.5	18.0	25.5
8GM " "		8.5	12.5	16.5
4X2 " " "		14.5	33.5	60.5
4X3 " "		19.5	47.0	88.0
4XG " "		16.5	41.0	77.0
GEC-120-8X0-792033		8.0	17.0	29.5
GEC-130-4X0-792033		27.5	61.0	105.0
GEC-130-DF2-792033		11.5	16.5	21.0
GEC-145-400-792033		40.0	82.5	146.0
GEC-140-800-792033		10.5	15.5	20.5
GEC-130-8X0-792033	*	8.0	14.0	20.5
GEC-140-4GX-792033		27.5	64.0	116.0
GEC-120-8A0-792033		8.5	13.5	17.0
GEC-130-8A0-792033		14.5	20.0	25.0
GEC-120-4A0-792033		30.5	64.0	110.0
GEC-130-4A0-792033		34.0	73.0	127.0
GEC-120-X40-792033	Feb	25.5	54.0	90.0
GEC-140-8G0-792033	Feb	17.0	24.5	31.0

TABLE 149. CALCULATED SPECIFIC GRAVITY AND API GRAVITY FOR TESTED FUELS

Sample designation	Specific gravity, 60/60°F	API gravity, °
GEC-131-DF2	0.8486	35.25
GEC-145-400	0.7607	54.51
GEC-140-800	0.8155	42.01
GEC-130-8XO	0.8268	39.64
GMSO F Farm Annex	0.8820	28.90
2040 Solvent B-19	0.9760	13.48
Xylene bottoms, Bldq. 42D	0.8718	30.81

TABLE 150. HEAT OF COMBUSTION OF TESTED FUELS

		Gross, B	tu/lb	Net, Btu/lb
Sample designation	Dupli	cates	Average	Average
JP-8 (2006), 78-C	19,785	19,782	19,784	18,514
8A2 " " "	19,124	19,144	19,134	18,045
8A3 " "	19,512	19,469	19,491	18,311
JP-4 (2006), 78-C	20,023	20,031	20,027	18,719
4A2 " "	19,207	19,209	19,208	18,114
4A3 " "	19,554	19,518	19,536	18,356
8X2 " "	19,187	19,196	19,192	18,119
8X3 " "	19,438	19,475	19,457	18,309
8GM 1: 0	19,809	19,791	19,800	18,556
4X2 " "	19,242	19,226	19,234	18,149
4X3 " " " " " " " " " " " " " " " " " " "	19,548	19,538	19,543	18,371
4XG " "	19,899	19,877	19,888	18,619
GEC-120-8X0-792033	19,203	19,169	19,186	18,088
GEC-130-4X0-792033	19,648	19,626	19,637	18,455
GEC-130-DF2-792033	19,465	19,501	19,485	18,307
GEC-145-400-792033	20,089	20,086	20,088	18,767
GEC-140-800-792033	19,845	19,851	19,848	18,576
GEC-130-8X0-792033	19,516	19,530	19,523	18,335
AR79029, GMSO F Farm Annex	19,953	19,950	19,952	18,662
Xylene Bottoms, Bldg. #42D	18,641	18,631	18,636	17,714
2040 Solvent B-19	17,945	17,935	17,940	17,186
CEC-140-4GX-792033	19,939	19,983	19,961	18,685
GEC-120-8A0-792033	19,201	19,244	19,223	18,137
GEC-130-8A0-792033	19,531	19,545	19,538	18,358
GEC-120-4A0-792033	19,294	19,262	19,278	18,189
GEC-130-4A0-792033	19,565	19,537	19,551	18,390
GEC-120-X40-792033 Feb	19,285	19,336	19,311	18,209
GEC-140-8G0-792033 Feb	19,826	19,811	19,819	18,549

GAS CHROMATOGRAPHIC SIMULATED DISTILLATIONS OF TESTED FUELS TABLE 151.

																	,	Feb	 	2	7		2	7	2	7	0	₹*	7	_	2	0	a	_	(continued)
X	S I	248	282	797	311	329	333	343	372	406	432	459	493	5.18	563	583	GEC-140-	2033	0	56	39	35	37.	39	41	43	45	46	48	51	25	29	649	67	onti
α	ပ္စ	120	139	147	155	165	167	173	189	208	222	237	256	270	595	306	235	8G0-792033 Feb	ບໍ	128	145	177	189	203	213	222	232	240	253	566	289	310	343	355	9
4/3	J. J.													258 496			3.20-	033 Feb	o E	88	111	196	223	284	315	331	334	338	345	358	430	475	558	581	
; 	 <u> </u>																GEC-3.20	X40-792033	ည	31	44	91	106	140	157	166	168	170	174	181	221	246	292	305	
4A2														538					1 . 1	_		_						_							
	8	35	44	86	104	130	153	183	209	223	243	248	267	281	304	314		4XG	P														628		
-4	- 6- E	70	72	142	176	203	232	259	289	334	374	410	446	473	505	514		7	ပြ	26	35	70	91	117	128	152	168	187	212	236	273	305	331	339	
dt.	ပ													245				4X3	oF.														3 532		
	أشا	4	2	2	_	9	9	7	9	-4	m	ر ک	9	4	6	2			•	Ř	4	æ	10;	129	14(164	17	18(19.	216	24	25.	278	787	
RAR	ို													514					6 F1	77	95	98	96	55	68	11	125	133	142	174	137	171	527	47	
	8	101	124	163	177	191	202	211	219	227	234	246	258	268	287	296		4X2	ပ														275 5		
RA?	e F																	æ	o F																
	ြိ	108	128	164	179	192	203	212	223	227	234	248	260	269	290	296		WE SE	ပ	129	143	169	183	197	208	217	227	235	247	259	281	302	325	331	
TP-8	o F	282	306	352	376	403	419	435	448	464	477	200	522	538	267	577		2	F	262	280	306	320	331	358	388	410	428	448	469	496	518	549	563	
	ပ	129	152	178	191	206	215	224	231	240	247	260	272	281	297	303		8X3	ပ	128	138	152	160	166	181	198	210	220	231	243	258	270	287	295	
Percent	recovered	0.5, IBP	1	2	70	20	30	40	20	09	70	80	06	95	66	99.5, FBP		Percent	recovered	0.5, IBP		5	10	20	30	40	20	9	70	80	06	95	66	99.5, FBP	

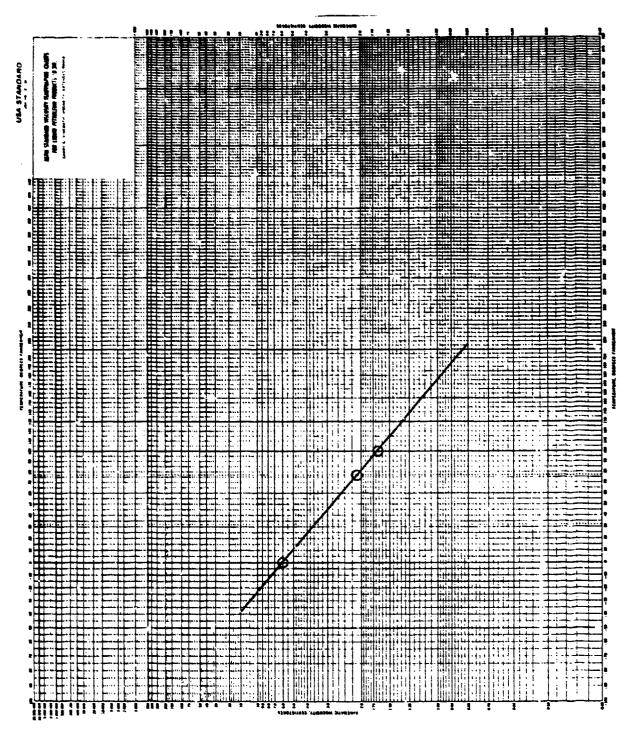
TABLE 151 (continued)

7 2		165 329 171 340 187 369 202 396 215 419 223 433		
7 2		190 374 203 397 214 417 222 432 231 448 239 462 249 480		GEC-130- 4A0-792033 "C "F" 30 86 37 99 79 174 96 205 121 250 138 280 164 327 187 369 206 403 223 433 232 450 253 487 264 507 291 556
GEC-145- 400-792033	25 77 34 93 72 162 93 199	93 199 108 226 123 253 141 286 160 320 180 356	218 424 237 459 254 489 273 523 281 538	GEC-120- 4A0-792032 C C F 34 93 47 117 88 190 103 217 129 264 160 320 186 367 208 406 221 448 237 459 255 491 271 520 296 565
GEC-130- DF2-792033	132 270 147 297 180 356 197 387	197 387 216 421 231 448 245 473 256 493 271 520 284 543	300 572 318 604 333 631 343 649 373 703	GEC-130- 8A0-792033 °C °F 136 277 149 300 176 349 189 372 201 394 211 412 219 426 228 442 219 426 228 442 228 442 228 253 242 468 253 487 266 511 275 527 296 565
GEC-130- 4 X0-792033		101 214 123 253 145 293 161 322 166 331 170 338		GEC-120- 8A0-792033 C °F 133 271 149 300 177 351 190 374 203 397 212 414 221 430 234 453 243 469 254 489 257 513 277 531 298 568 304 579
GEC-120- 8X0-792033	80 176 97 207 144 291			GEC-140- 4GX-792033 °C °F 28 82 36 97 78 172 98 208 121 250 143 289 164 327 178 352 204 399 230 446 264 507 301 574 318 604 342 648 346 655
Percent recovered	0.5 IBP 1 5	20 30 50 50 70	80 90 95 99 99.5 FBP	Percent recovered 0.5 LbP 1 5 10 20 30 40 50 60 70 80 90 99 99.5 FBP

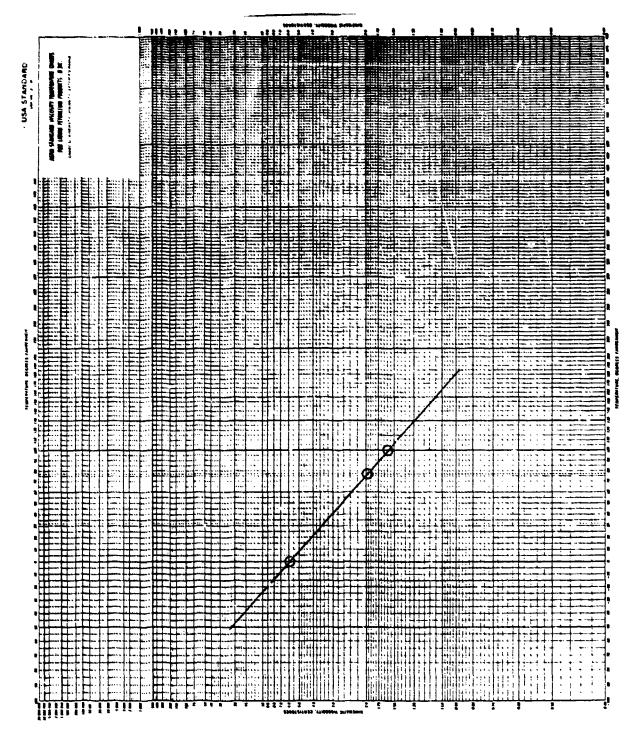
HYDROCARBON-TYPE DISTRIBUTION OF TESTED FUELS (ASTM D 2789) TABLE 152.

	ļ					Ligu	Liquid volume percent	me pe	rcent				
Compound type	JP-8	8A2	8A3	JP-4	4A 2	4A3	8X2	8X3	8GM	4X2	4X3	4XG	GEC-120- 8XO-792033
Paraffins	42.3	29.1	35.9	62.2	37.6	45.6	23.8	33.0	43.9	30.6	44.4	55.3	27.0
Monocycloparaffins	41.0	28.0	34.4	24.1	20.5	25.0	20.5	30.2	39.1	10.4	15.1	25.4	22.6
Dicycloparaffins	3 1	1.9	5.6	3.4	0.0	0.0	2.3	3.1	3.3	2.4	3.7	1.1	1.0
Alkylbenzenes	7.5	11.3	9.4	8.4	12.6	11.0	52.4	30.3	7.6	55.6	35.3	16.2	48.9
Indans and tetralins	4.1		5.2	1.3	4.5	3.3	0.0	1.8	4.1	9.0	1.0	1.4	0.0
Naphthalenes	2.0	23.7	12.5	9.0	24.8	15.1	1.0	1.6	2.0	4.0	0.5	9.0	0.5
						Liqu	Liquid volume		percent				
	GEC-130-	130-	GEC-130-	130-	GEC-140-	140-	GEC-145-		GEC-130-	130-	GEC	GEC-120-	GEC-140-
Compound type	4X0-7	4X0-792033	8X0-792033	92033	800-792033	92033	400-792033	92033	DF2-7	DF2-792033 ^a	4X0-7	4X0-79203	4GX-792033
Paraffins	45	rv.	36.2	ci	44.4	4.	61	8	49.1	Η.	62.	&,	59.1
Monocycloparaffins	18	w.	33.4	4.	41.4	4	24.2	7	26.7	.7	25.1	-	24.0
Dicycloparaffins	ĸ	3.3	2	2.3	7	2.6	4	6.		q-	4.6	بو	4.4
Alkylbenzenes	32	7.	25.4	4.	ý	6.7	80	8.2	10	بو	6.6	ور	11.8
Indans and tetralins	0	0.0	1	z.	m	4.	-	.1	4	9.	0	6.	0.7
Indenes and Dihydro-													
naphthalenes				1	•		•		0	8.0	•		•
Naphthalenes	0	0.2	1	.2	-	ı.	Ö	0.4	80	.2	<0.1	-	<0.1
						Liquid	id volu	volume percent	rcent				
											GEC-120-	120-	GEC-140-
	GEC-140-	140-	GEC-120-	120-	GEC-130-	130-	GEC-120-	-071	GEC-130	-0£1	X40-792033	32033	8G0-792033
Compound type	8G0-7	8G0-792033	8A0-792033	92033	8A0-792033	32033	4A0-792033	32033	4A0-792033	92033	Feb		Feb
Paraffins	41	41.4	35.7	.7	41.2	.2	42.4	4.	48	หา	36.	.2	47.2
Monocycloparaffins	38	.2	34	34.8	40.0	0.	28.1	-	32.0	0	15.0	0	42.7
Dicycloparaffins	-	1.6	9	6.3	0	₽.	0	0.	0	0.	2	9.	1.0
Alkylbenzenes	15	6.	σ	6.6	7	.7	11	.2	\$	۳.	45.	6.	0.9
Indans and tetralins	2	2.0	4	4.	m	3.4	m	3.0	-	9	Ö	د .	
Naphthalenes	0	6.	14	6.	7	ε.	15	ε,	80	9.	o	0.	0.7

 $^{^{}m b}$ Dash indicates that the compound type is not included in the analysis. Monsanto Method 21-PQ-38-53 used for this analyses.

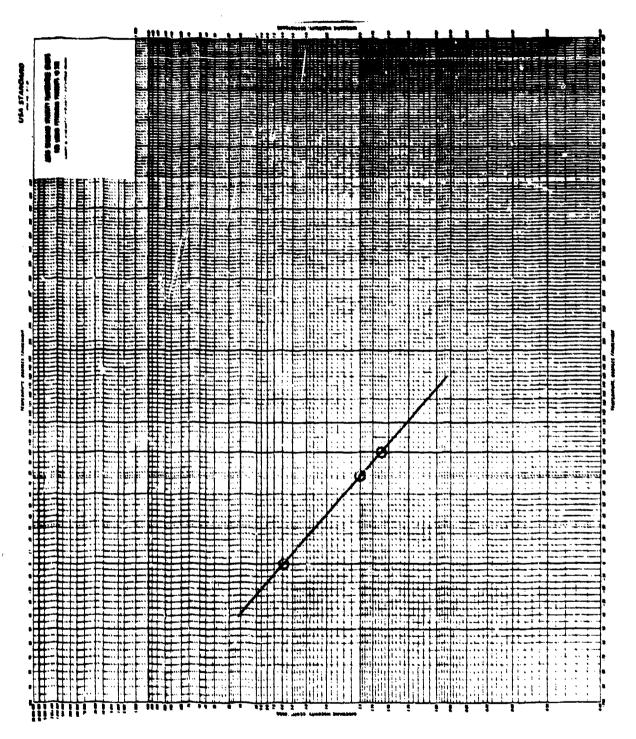


Viscosity/temperature plot for JP-8 fuel, (2006),78C. Figure 109.

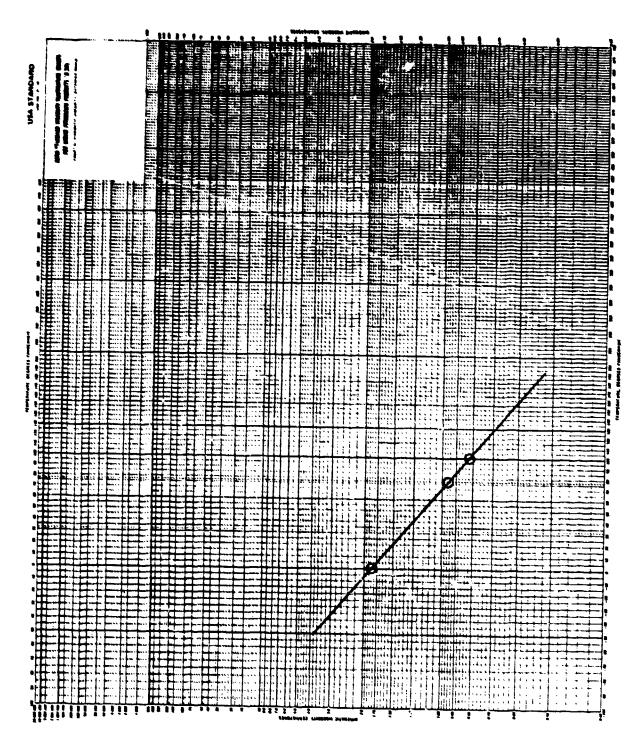


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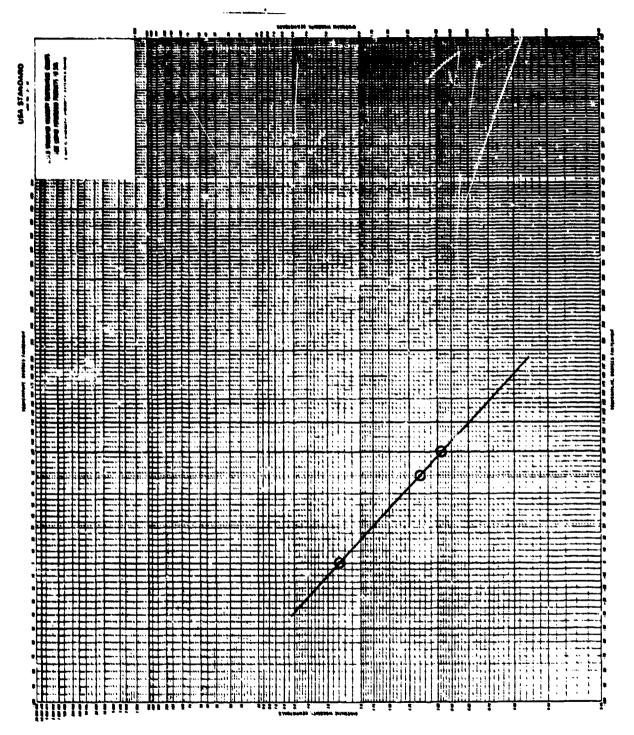
Viscosity/temperature plot for fuel sample 8A2 Figure 110.



Viscosity/temperature plot for fuel sample 8A3. Figure 111.

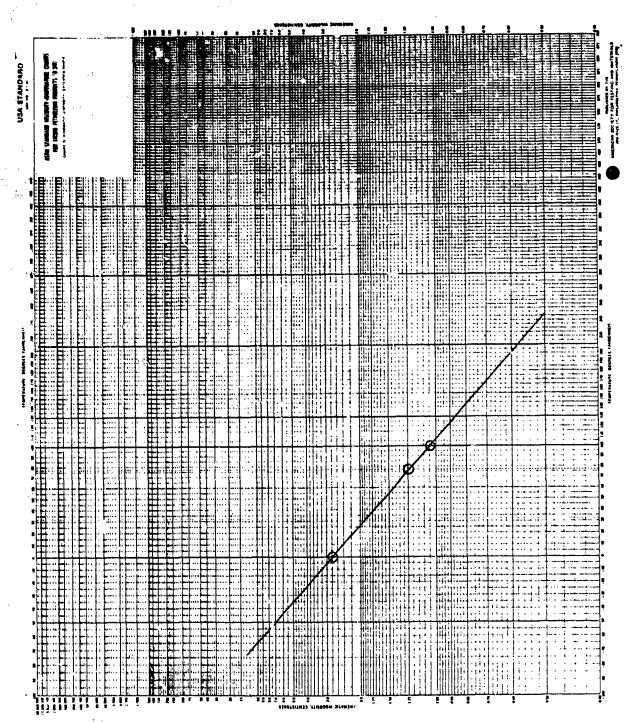


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Viscosity/temperature plot for JP-4 fuel, (2006),78C.



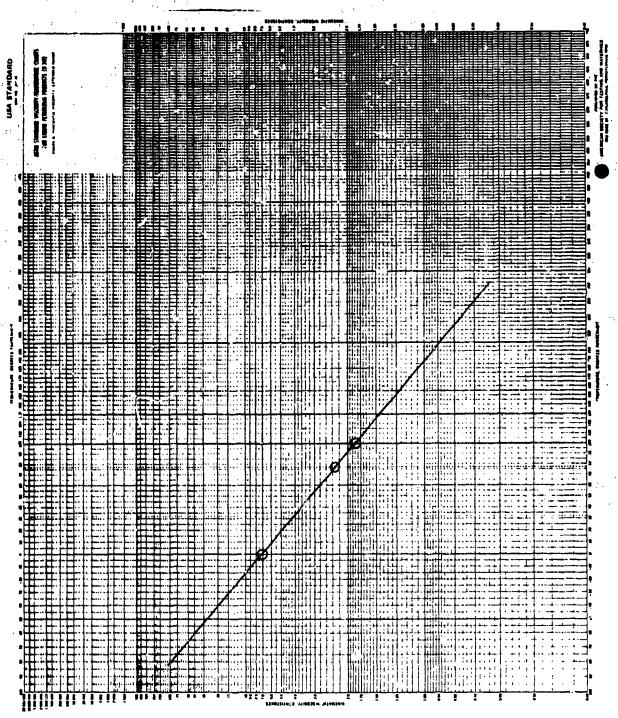
Viscosity/temperature plot for fuel sample 4A2. Figure 113.

Viscosity/temperature plot for fuel sample 4A3 Figure 114.



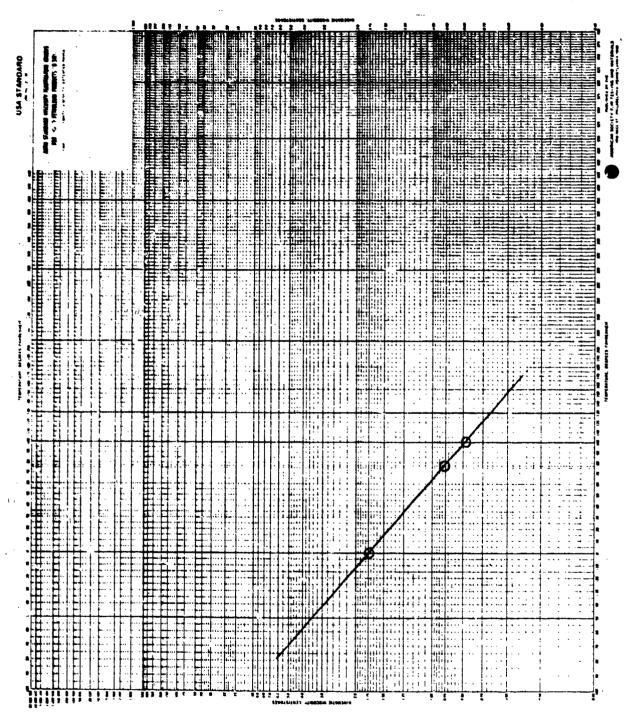
Viscosity/temperature plot for fuel sample 8X2. Figure 115.

Viscosity/temperature plot for fuel sample 8X3 Figure 116.

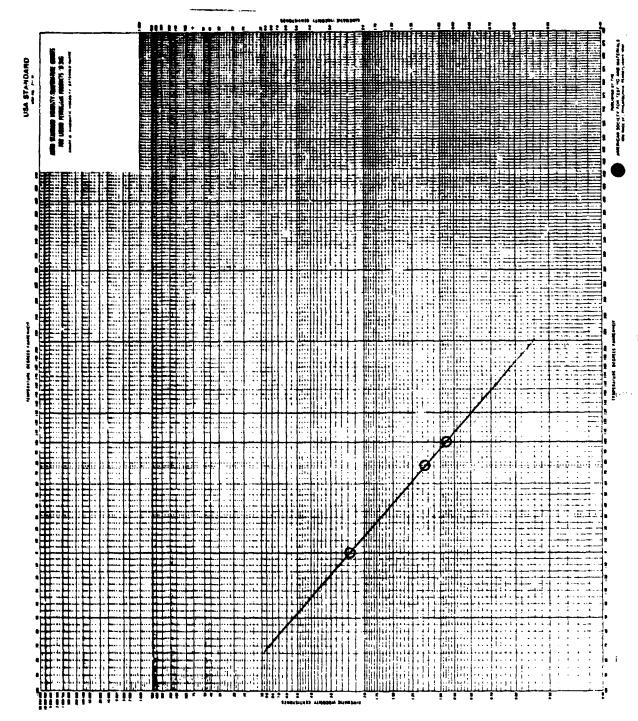


Viscosity/temperature plot for fuel sample 8GM

Figure 118. Viscosity/temperature plot for fuel sample AX2

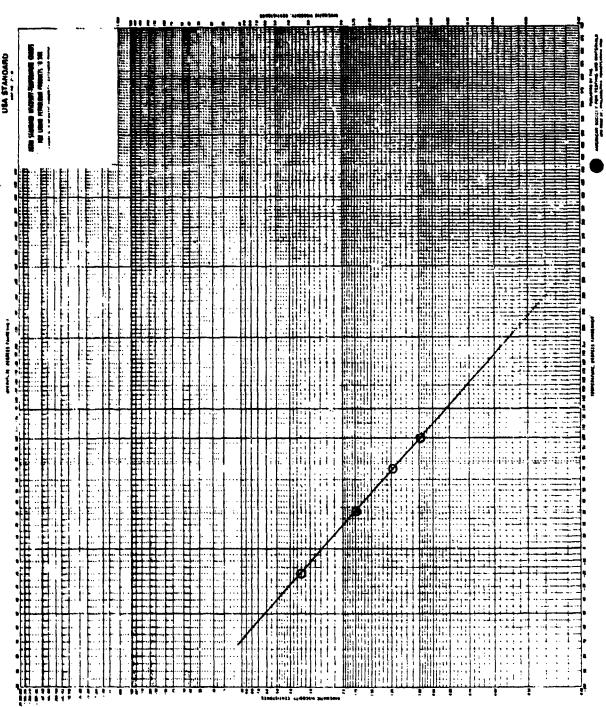


Viscosity/temperature plot for fuel sample 4X3. Figure 119.



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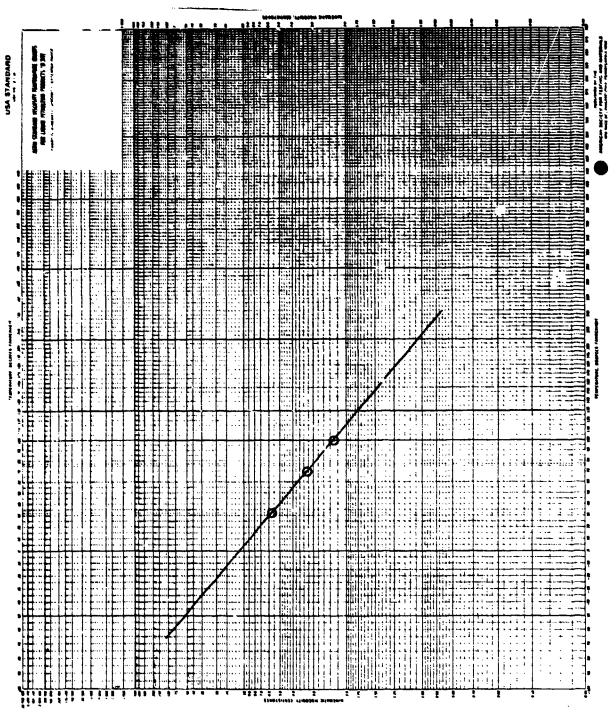
Figure 120. Viscosity/temperature plot for fuel sample 4XG



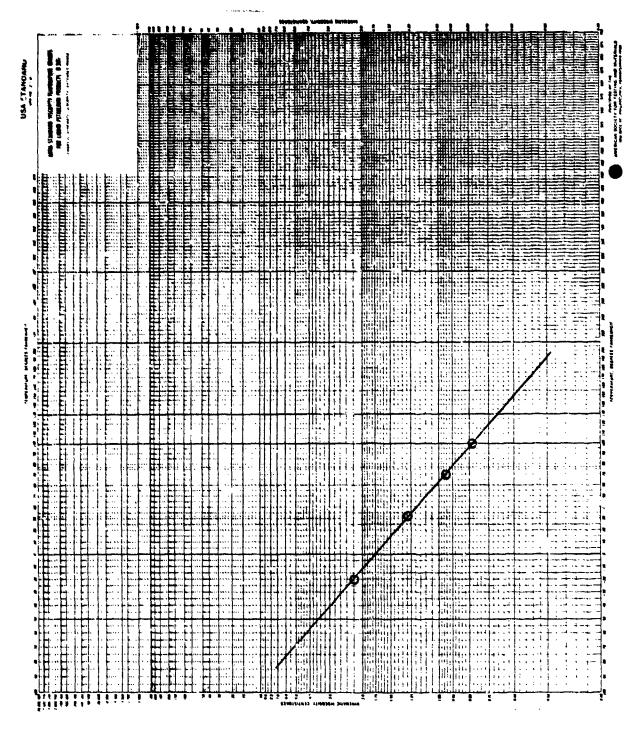
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Viscosity/temperature plot for fuel sample GEC-120-8X0-792033. Figure 121.

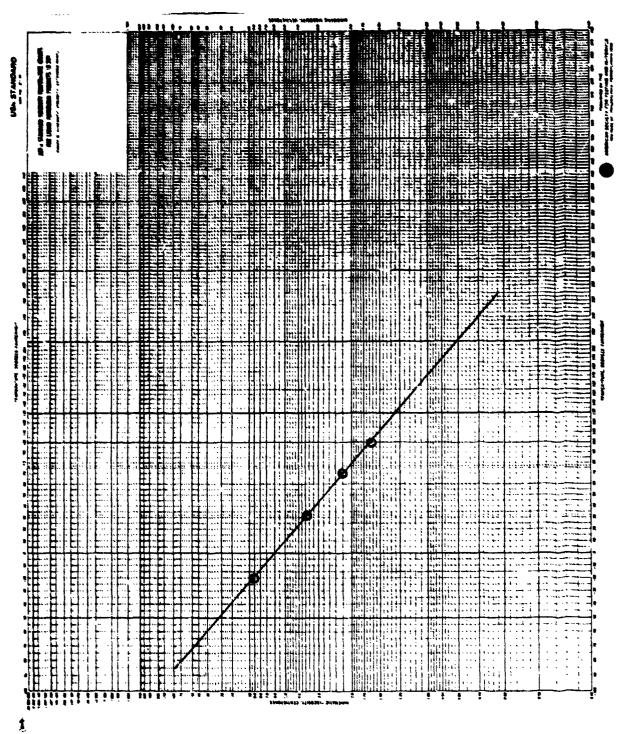
Figure 122. Viscosity/temperature plot for fuel sample GEC-130-4X0-792033.



Viscosity/temperature plot for fuel sample GEC-130-DF2-792033 Figure 123.

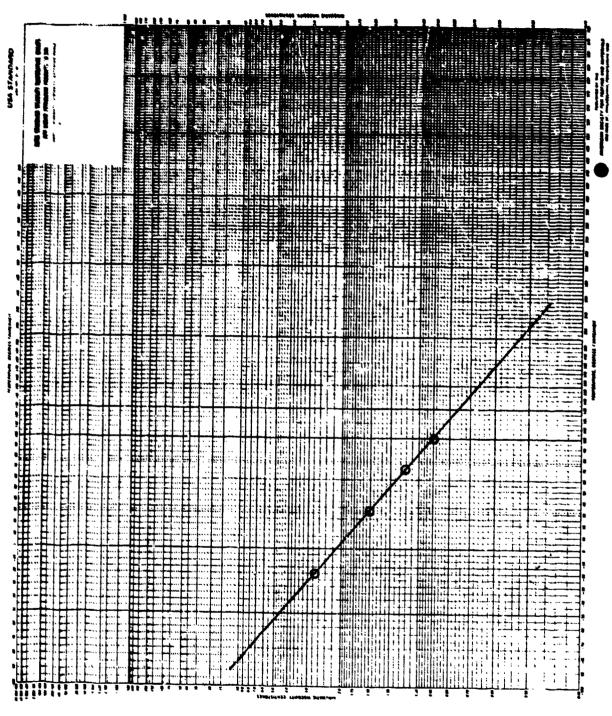


Viscosity/temperature plot for fuel sample GEC-145-400-792033 Figure 124.



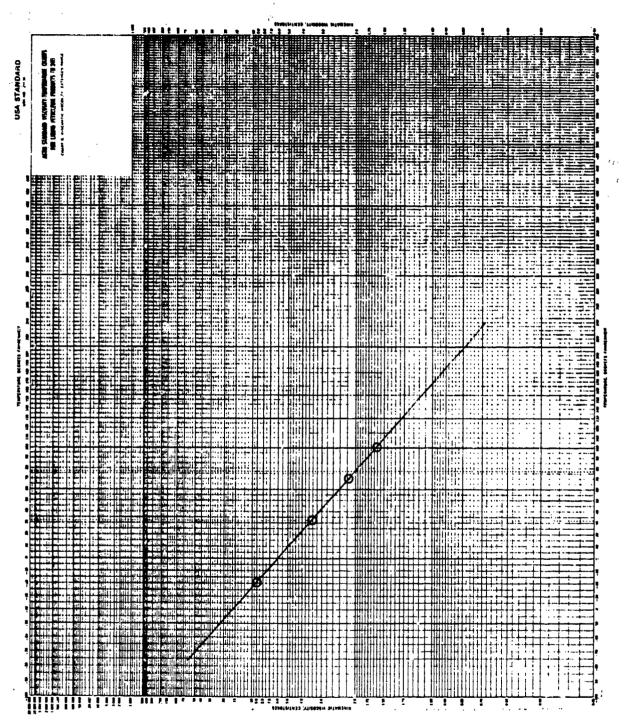
Viscosity/temperature plot for fuel sample GEC-140-800-792033 Figure 125.

Viscosity/temperature plot for fuel sample GEC-130-8X0-792033 Figure 126.



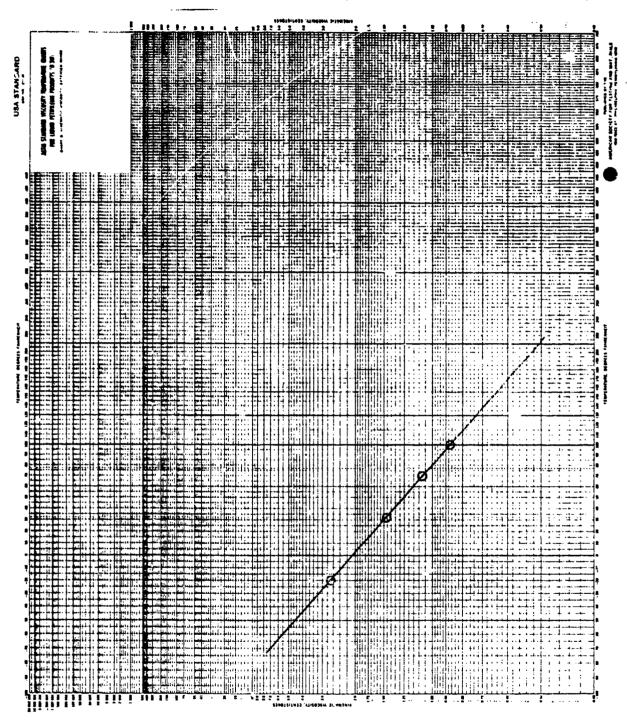
Viscosity/temperature plot for fuel sample GEC-140-4GX-792033. Figure 127.

Viscosity/temperature plot for fuel sample GEC-120-8AO-792033 Figure 128.



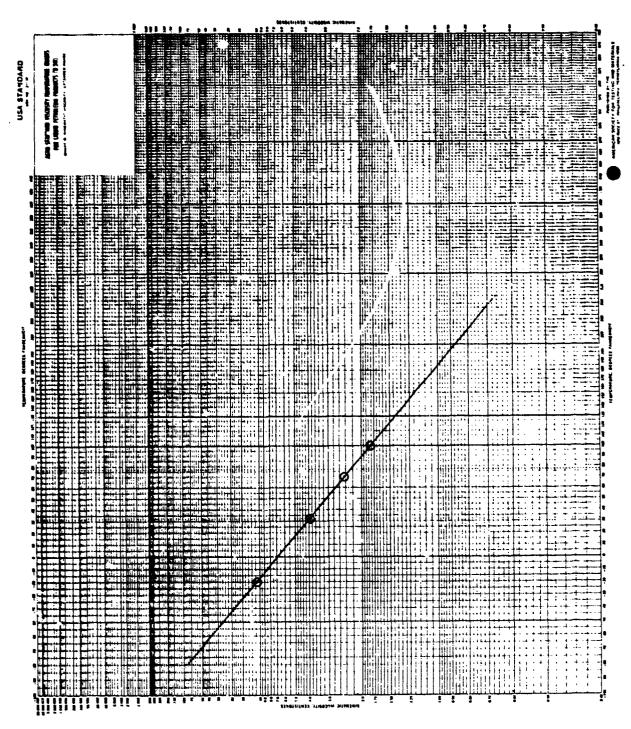
Viscosity/temperature plot for fuel sample GEC-130-8A0-792033 Figure 129.

Viscosity/temperature plot for fuel sample GEC-120-4A0-792033 Figure 130.



Viscosity/temperature plot for fuel sample GEC-130-4A0-792333 Figure 131.

Viscosity/temperature plot for fuel sample GEC-120-X40-792033 Feb



Viscosity/temperature plot for fuel sample GEC-140-8GO-792033 Feb. Figure 133.

first number in the codes for these fuels indicates whether the base fuel was JP-4 (4) or JP-8 (8). The letter(s) following the first number indicate whether the blending stock was 2040 Solvent (A), xylene bottoms (X), or Gulf Mineral Seal Oil (GM). number in the initial three characters of the fuel code indicates the nominal hydrogen content 12% (2) or 13% (3). The fuel codes terminating with "792033" were tested in the J79 Low-Smoke Combustor; sample codes for these fuels are as follows. following the first hyphen now designates the nominal blended hydrogen content (12.0%, 13.0%, or 14.0%). The number and letters indicate the fuel type (JP-4 or JP-8) and the blending stock. Aromatic 2040 stock only (AO), xylene bottoms only (XO), Gulf Mineral Seal Oil only (GO), and Gulf Mineral Seal Oil and xylene bottoms (GX) are the blending materials used. All fuels were provided to MRC after blending.

10. PHYSICAL PROPERTIES OF EXPERIMENTAL FUEL BLENDS

the accord towards towards so

A TRADESCRIPTION OF SECURITION
Before-and-after testing property data were required for a number of experimental fuel blends which had been subjected to full-scale testing by an engine manufacturer. The engine manufacturer's tests included idle point, ignition, and fouling. The physical property measurements were conducted by the procedures described in the Appendix, and the analytical data are shown in the following tables:

Kinematic viscosity by ASTM D 445 - Table 153 and Figures 134-165,
Vapor pressure by the micro-method - Table 154,
Density by the dilatometer method - Table 155,
Specific gravity and API gravity from density data - Table 156,
Surface tension by the capillary rise method - Table 157,
Fydrocarbon type by ASTM D 2789 - Table 158,
Simulated distillation by gas chromatography, ASTM D 2887 - Table 159,

Specific gravity and API gravity were calculated from the density data shown in Table 155.

TABLE 153. KINEMATIC VISCOSITY OF EXPERIMENTAL FUEL BLENDS

	(centi stoke	es
Sample	0°F	77°F	100°F
Fouling test			
#1	1.866	0.9315	0.8035
#2	2.601	1.146	0.9665
#4	1.703	0.8860	0.7654
#5	1.765	0.9146	0.7890
#6	2.370	1.133	0.9627
#7	5.974	2.062	1.657
#8	5.793	1.956	1.572
#9	5.834	1.999	1.609
#10	2.750	1.231	1.034
#12	7.105	2.307	1.833
_ ,,,			
Ignition			
#1, JP-4	1.934	0.9796	0.8407
#2	2.825	1.243	1.044
#3	2.430	1.128	0.9562
#5	2.753	1.240	1.043
#6	2.480	1.161	0.9851
#7, JP-8	5.441	1.942	1.571
#8	5.711	1.938	1.560
#9	5.223	1.864	1.510
#10	2.662	1.209	1.018
#11	3.504	1.450	1.203
#12	3.918	1.580	1.303
rus 1 dala madma			
Fuel idle point #1	1.857	0.9494	0.8185
#1 #2	2.609	1.171	0.9880
#2 #3	2.218	1.062	0.9053
#3 #4	1.716	0.8908	0.7685
	1.718	0.8908	0.7879
#5 • 7			
# 7	6.053	2.075	1.667
#8	5.819	1.956	1.572
#9	5.873	2.003	1.611
#10	2.715	1.228	1.034
#11	3.740	1.516	1.253
#12	7.090	2.303	1.829

TABLE 154. VAPOR PRESSURE OF EXPERIMENTAL FUEL BLENDS

Sample		32°F	mm HG	100°F
<u> </u>		3	<u> 70 E</u>	100 F
Fouling Test	#1 #2	41.0 20.5	86.0 47.5	146.0 86.0
	#4	21.5	45.5	77.0
	# 5	20.5	47.5	86.0
	#6	30.5	66.0	114.0
	#7	9.5	15.0	20.5
	#8	11.0	17.0	27.5
	49	10.0	16.5	27.5
	#10	7.0	13.5	21.5
	#12	11.5	16.0	21.0
Ignition	#1, JP-4	29.5	65.0	113.0
	#2	19.5	44.0	76.5
	#3	22.0	48,0	83.0
	# 5	17.0	39.5	69.0
	# 6	22.0	51.0	91.0
	#7. JP-8	11.0	20.5	31.0
	#8	9.5	16.0	22.5
	#9	10.0	19.0	29.5
	#10	12.0	26.0	45.0
	#11	9.5	20.5	34.0
	#12	13.0	26.0	42.0
Fuel	#1 ,	41.5	86.0	143.0
	#2	21.0	48.5	86.0
•	#3	28.5	63.5	111.0
	#4	17.5	39.0	67.5
	# 5	24.0	53.5	93.5
	47 ,	10.0	14.5	18.5
	#8	10.5	16.0	22.0
	#9	7.0	11.5	16.5
	#10	5.0	10.5	19.0
	#11	5.5	11.0	17.5
	#12	10.0	14.5	19.5

TABLE 155. DENSITY OF EXPERIMENTAL FUEL BLENDS

		Crame no	Grams per cubic centimeter											
			r cubic cent	ımeter										
Sample		32°F	70°F	100°F										
Ignition	#1	0.7769	0.7603	0.7474										
Ignition	#2	0.8449	0.8285	0.8154										
Ignition	#3	0.8296	0.8130	0.8001										
Ignition	#5	0.8230	0.8068	0.7941										
Ignition	#6	0.7952	0.7795	0.7669										
Ignition	#7	0.8214	0.8066	0.7946										
Ignition	#8	0.8709	0.8552	0.8430										
Ignition	#9	0.8443	0.8288	0.81.66										
Ignition	#10	0.8424	0.8261	0.8132										
Ignition	#11	0.8395	0.8239	0.8112										
Ignition	#12	0.8286	0.9128	0.8004										

TABLE 156. GRAVITY VALUES FOR EXPERIMENTAL FUEL BLENDS

Sample		Specific Gravity 60/60°F	API Gravity,°
Ignition	#1	0.765	53.47
Ignition	#2	0.834	38.16
Ignition	#3	0.813	41.48
Ignition	#5	0.812	42.76
Ignition		0.784	48.98
Ignition		0.811	42.98
Ignition		0.860	33.03
Ignition		0.834	38.16
Ignition		0.831	38.78
Ignition		0.829	39.19
Ignition		0.818	41.48
-			

TABLE 157. SURFACE TENSION OF EXPERIMENTAL FUEL BLENDS

		Dynes/cm	
Sample	32°F	<u>70°F</u>	100°F
Ignition #1	25.81	23.67	22.01
Ignition #2	28.09	26.28	24.86
Ignition #3	27.38	25.93	24.08
Ignition #5	27,98	25.52	24.35
Ignition #6	.i.61	24.67	23.17
Ignition #7	28.91	26.89	25.31
Ignition #8	30.47	28.53	27.02
Ignition #9	29.50	27.33	25.63
Ignition #10	28.70	26.80	25.35
Ignition #11	29.09	27.00	25.38
Ignition #12	28.78	26.75	25.18

AND VARIOUS WARREST MARKET TO THE WARREST WARREST WARREST TO AND THE SECOND WARREST TO THE WARREST WARREST TO THE WARREST WARR

TABLE 158. HYDROCARBON-TYPE ANALYSIS OF EXPERIMENTAL FUEL BLENDS FROM IGNITION TESTS

					Liquid	Volume	Percent				
Compound Types	41	#2	43	\$5	16	17	₩8	#9	#10	111	112
Paraffins Monocycloparaffins Dicycloparaffins Alkylbenzenes Indans & Tetralins Naphthalenes	63.8 22.0 4.8 7.7 1.3	40.1 25.2 16.6 2.1 15.0	42.0 25.4 - 22.1 1.7 8.8	43.5 29.6 21.0 1.5 4.4	58.1 21.3 5.5 13.2 1.4 0.5	45.1 41.4 1.8 7.0 3.3	33.4 30.8 1.6 10.7 5.2 18.3	39.8 35.4 1.5 9.5 4.1 9.7	32.9 24.8 0.3 37.3 0.3 4.4	37.8 30.8 0.8 24.7 1.8 5.1	41.5 33.4 0.8 20.9 1.7

SIMULATED DISTILLATION BY GAS CHROMATOGRAPHY TABLE 159.

	 										ı	lan I										Ja 1			;	4	7-1 ₁ -					
	Fouling Test #4	52	138	216	253	291	297	340	403	493	Fouling Test 67	. 46	235	336	36.1	907	424	£\$ 9	4.5	509	559 574	Folling Test \$10	216	253	289	307	329	396	4 21	477	1 13 10 10 10 10 10 10 10	1
;	Fouling BP, °C	11	9	102	123	144	147	171	206	72 E	rouling.	2. '49	113	170	184 100	508	225	234	253	265	293 301	Fouling	102	123	143	153	165	202	216 229	247	282))
	Fouling Test #2 BP, "C BP, "F	72	167	234	277	376	4 4 6 6 8 8 8 8	77	6 7 8 8 0 8	547	Fouling Test #6	4. '49	8	167	196	/ /	338	376	419	531	635 649	Fouling Test #9 BP, *C BP, *P	252	282	367	392 410	428	455	4 68 4 91	513	995	i j
,	Pouling	22	75	112	136	191	206 224	229	248 262	286 295	Foultm	D. 7d6	27	27	32	134	155	191	237	277 205	335 343	Fouling BP, C	122	139	186	200 210	220	235	2 4 2 255	267	30¢	r I J
,	Fouling Test #1 BP, *C BP, *F	30	1 8 2 1 1 E	138	167	223	266 306	342	379 406	7 8	Foulthy Test #5	Bb, P	73	174	223 248	286	327	342	415	455	532 556	Fouling Test #8 BP, *C BP, *P	257	298	374	399	432	462	4 78	518	60 50 60 50 60 50 60 50	•
	Fouling	7 ;	28	6	75	901	152		208	229	Foul ing		6 6	79	120	141	164	172	213	235	278	Fouling BP, C	125	142	061	204	222	239	248 257	270	302	
	Recovered	0.5, IBP 1		20.	30 4 0	200	60	000	9 6 5 5	99 99.5, FBP	Percent	Recovered	0.5, IBP 1	د مه	70 70 70	000	20	097	2 08	06	99 99.5. FBP	Percent Recovered	0.5, IBP	~ ; v	10	30	0 0 0	09	08	90	99.5, FB2	

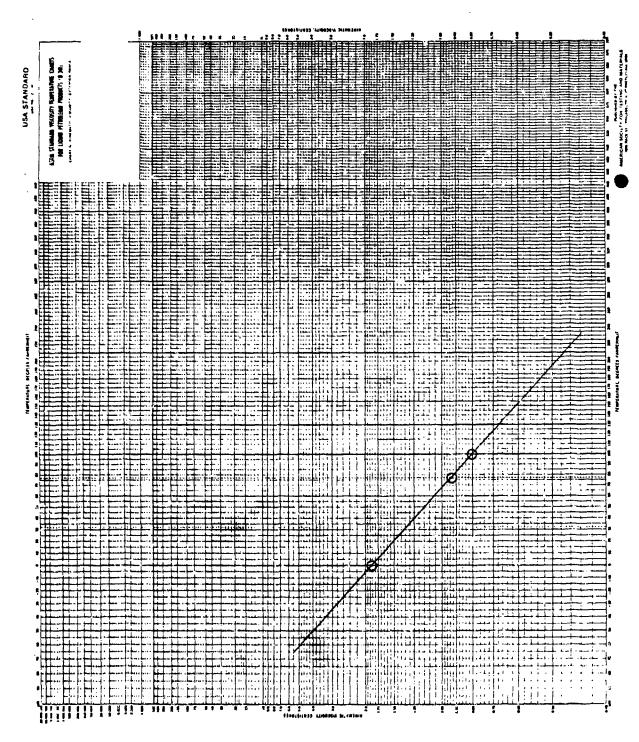
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Ignition 62 BP, of	37 49 120 115 115 143 175 175 376	213 228 244 441 253 487 271 520 285 307 585 317 603	Ignition 96 17 63 17 63 185 100 212 118 212 118 212 119 212 154 352 178 352
Ignition #1, JP-4	25 77 81 65 149 87 105 221 124 255 1141 286	165 1185 205 205 401 242 242 256 493 276 529 554	Ignition 65 33 91 53 127 91 228 109 228 163 325 173 343 193 379 210 410 212 459 225 493 272 581 317 603
Fouling Test #12 BP, *C BP, *F		231 448 240 464 251 484 290 554 307 595 336 637	Ignition 63 29 84 39 102 86 187 102 216 1128 262 1138 262 1189 313 169 313 206 493 256 693 258 514 292 518
Percent Recovered	0.5, IBP 1 5 5 10 20 30 40	50 60 80 90 95 99-5, 7 8P	Percent Recovered 0.5, IBP 1 10 20 30 40 50 60 60 60 90 99 95 99

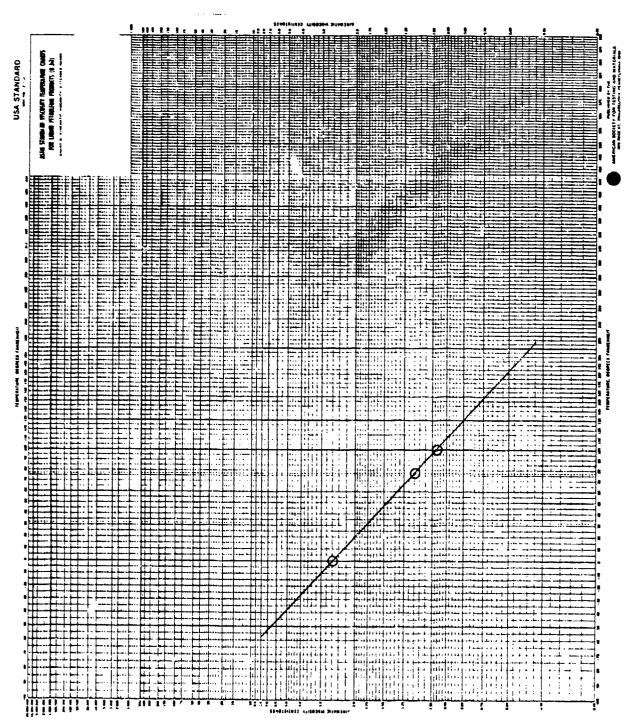
TABLE 159 (continued)

			m 1	i -	.
on 69 BP, °F	156 1194 286 338 379 401	6 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	Ignition \$12 57 135 69 155 18 244 45 293 65 3329		1 63 1P 15 61 15 6 15 6 15 6 15 6 17 7 23 9 23 9 24 1 25 1 26 1 27
Ignition 69	141 170 193 205	217 224 234 254 264 305 317	110 110 110 110 110 110		24 27 27 27 27 27 27 27 20 20 20 20 20 20 20 20 20 20 20 20 20
Ignition #8	18 225 313 342 390	8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	1gn/tion #11 5C BP, F 50 122 64 147 107 225 334 306 650 330		11 92 IP 12 95 12 95 13 95 14 95 15 95 16 95 17 95 18 95
Igniti	85 107 175 188 199	200 212 232 261 261 261 261 261	Igniti BP, 6C 50 64 107 134 152		Puel 26 35 35 1120 114 114 1199 2312 2312 244 259 306
67, JP-8 BP, 6F	167 203 3352 3852 406	7 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	190 194 194 194 194 194 194 194 194 194 194	5	Fuel #1, Idle Point 13
Imition #7,	75 95 1150 118 1198 208	225 225 225 225 225 226 226 226	Ignitis 11 50 90 11 11 11 11 11 11 11 11 11 11 11 11 11	100 100 100 100 100 100 100 100 100 100	Fuel 61, 1 13 13 13 102 136 131 131 131 131 131 131 131 131 131
Percent Recovered	0.5, IBP 10.5, IBP 10.00 10.00 10.00	560 560 90 90 90 90 90 90 70 70 70	Percent Recovered 0.5, IBP 10 5 10 30	60 50 80 90 995 788	Percent Recovered 0.5, IBP 1 10 30 40 50 60 60 90 99 995, FBP

die Point	259	340	290	390	904	717	451	997	583	209	525	900	208	Fuel #10 IP.	BP. *F	707	264	289	306	324	329	333	369	403	4 30		513	549	563				-											
Puel 67, Edle Point	126	171	104	661	208	225	233	241	253	265	274	667	A É 7	Fuel	35, °C	•1	129	143	152	162	165	170	107	50 6	717	25.6	267	287	295															
ruel 65 IP	75	156	198	244	//7	331	343	369	412	457	7	273		Puel 49 IP	BP, *P	198	353	334	361	,	406	423	439	451	7) G	529	574	929	1 #12 IP	46 '4B	270	291	342	7	412	430	770	462) (S	582	635	579
BP, C	7,5	2,2	92	811	154	166	173	187	211	236	251	786		Pue	BP, °C	4.2	123	163	183	198	208	217	226	233	747	365	276	301	330		3, 'da	132	77	172	900	217	221	230	239	169	+ 65 70 71 71 71	307	335	343
Fuel 64 IP BP, °F	8 1	174	207	757	293	333	340	352	390	877	4 62	0 Y		18 II	36, °F	241	275	347	370	396	† 1	430	450	424	107	516	536	576	986	911 IP	C BB, • F	277	289	315	677	35.6	392	414	433	477 777	504	325	559	572
Fue BP. C	27	6/	97	777	151	167	171	178	199	231	250	20.7	• • • • • • • • • • • • • • • • • • • •	Fuel	3, 'de	116	135	175	186	202	212	221	232	237	257	269	280	302	306		36, °C	136	143	157	27.	179	200	212	223	633	262	27.4	293	300
Percent Recovered	6.5, IBP	. •	91	07.	9 9	20	09	70	80	00.	٠ د د د د د د د د د د د د د د د د د د د	90.5	•	Percent	Recovered	0.5, IBP		•	10	20	30	4	000	700	. 6	06	95	66	99.5, FBP	Percent	Recovered	0.5, IBP	-	· ·	20	90	9	50	90	2 4	26	95		99.5, FEP

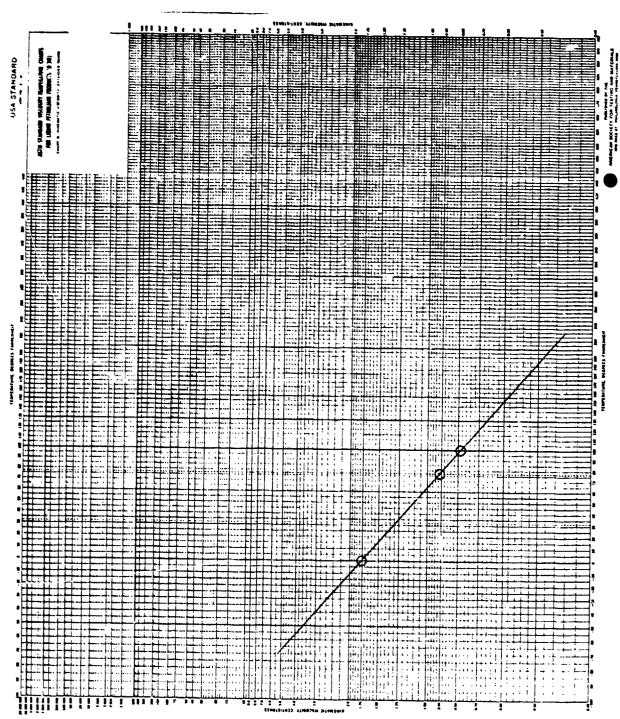


Viscosity/temperature plot for fuel sample from Fouling Test Figure 134.

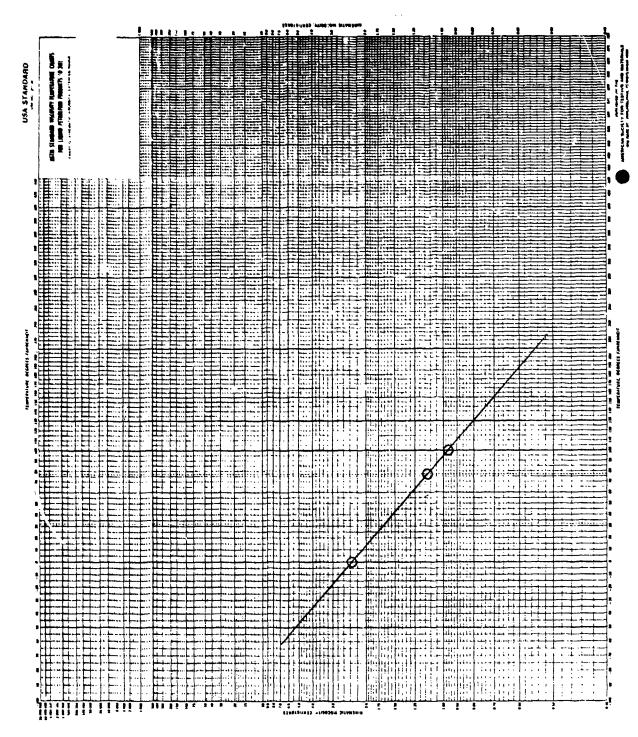


Viscosity/temperature plot for fuel sample from Fouling Test #2 Figure 135.

Viscosity/temperature plot for fuel sample from Fouling Test Figure 136.



Viscosity/temperature plot for fuel sample from Fouling Test #5. Figure 137.



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Figure 138. Viscosity/temperature plot for fuel sample from Fouling Test #6

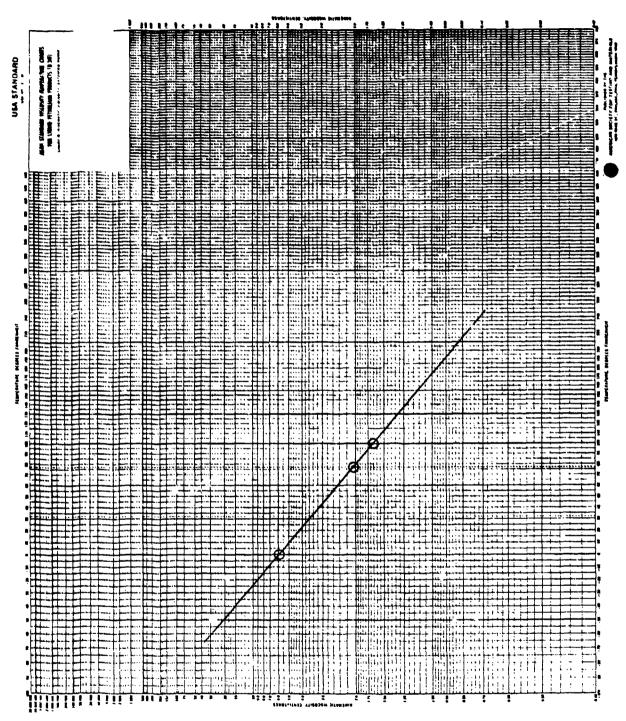


Figure 139. Viscosity/temperature plot for fuel sample from Fouling Test #7

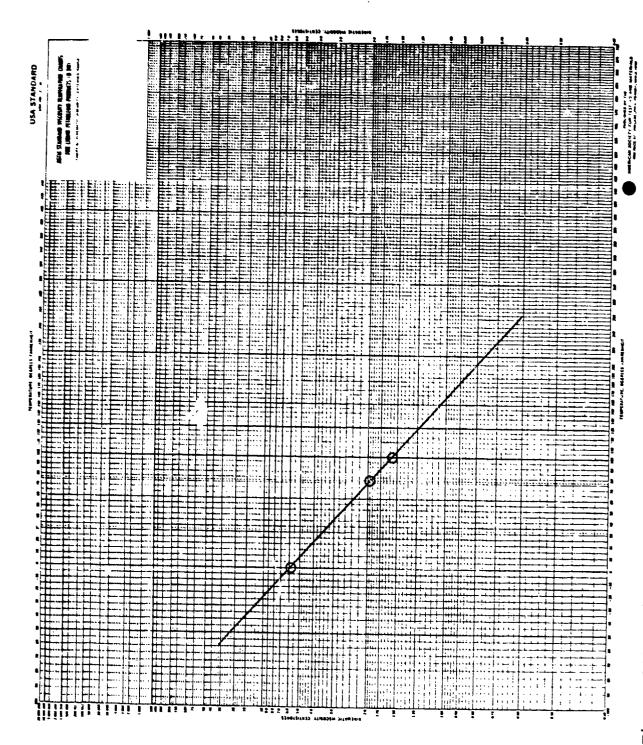
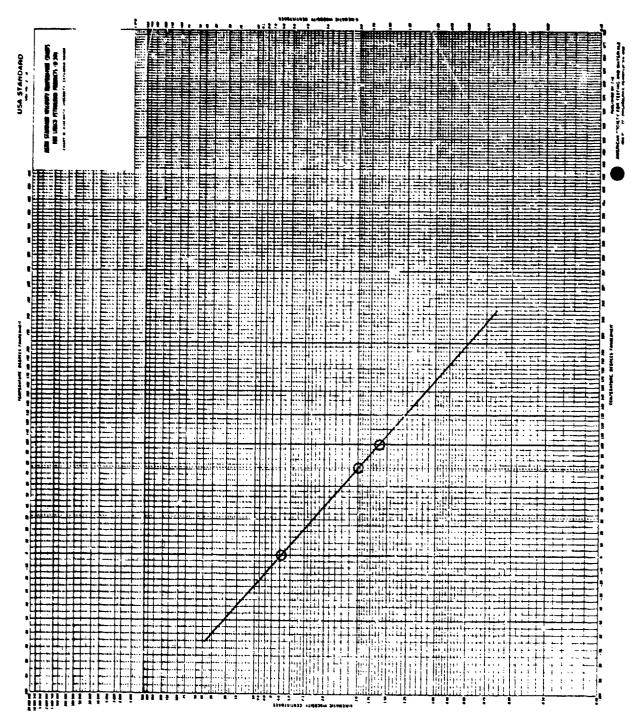
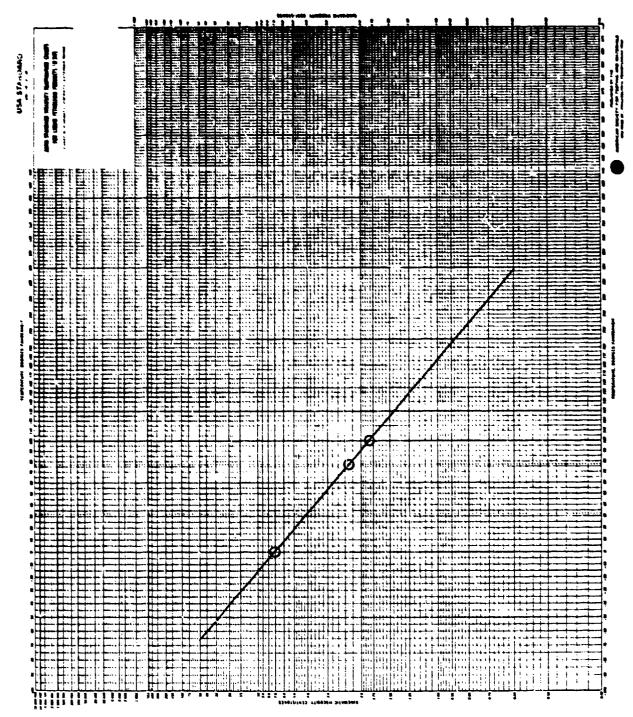


Figure 140. Viscosity/temperature plot for fuel sample from Fouling Test



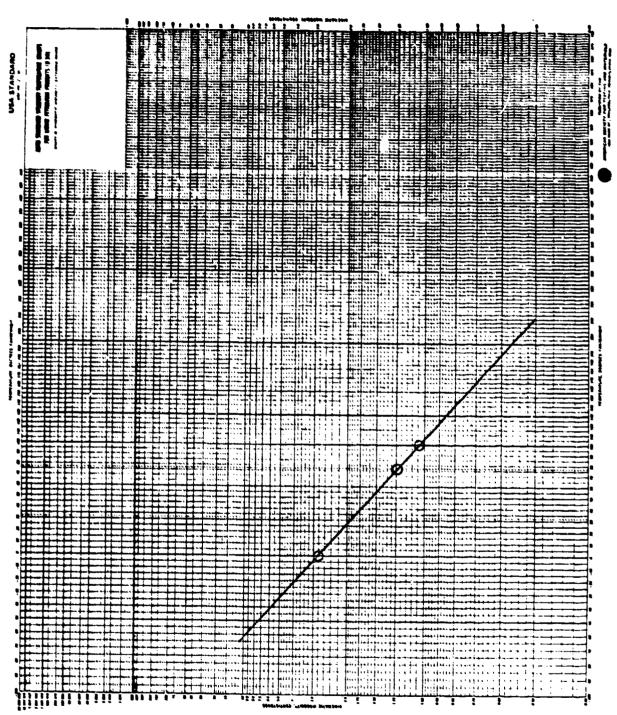
Viscosity/temperature plot for fuel sample from Fouling Test #9 Figure 141.

Viscosity/temperature plot for fuel sample from Fouling Test #10 Figure 142.



Viscosity/temperature plot for fuel sample from Fouling Test

Viscosity/temperature plot for fuel sample from Ignition #1, JP-4



Viscosity/temperature plot for fuel sample fror Ignition #2.

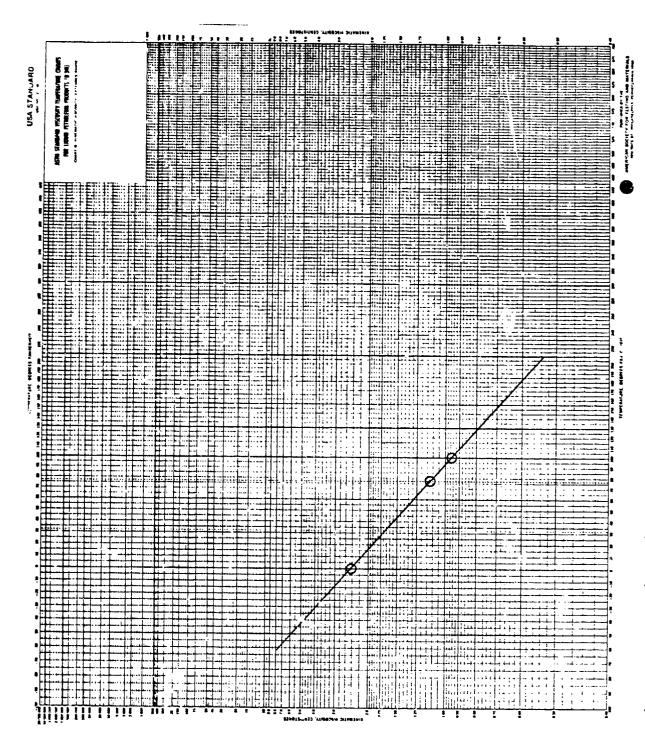
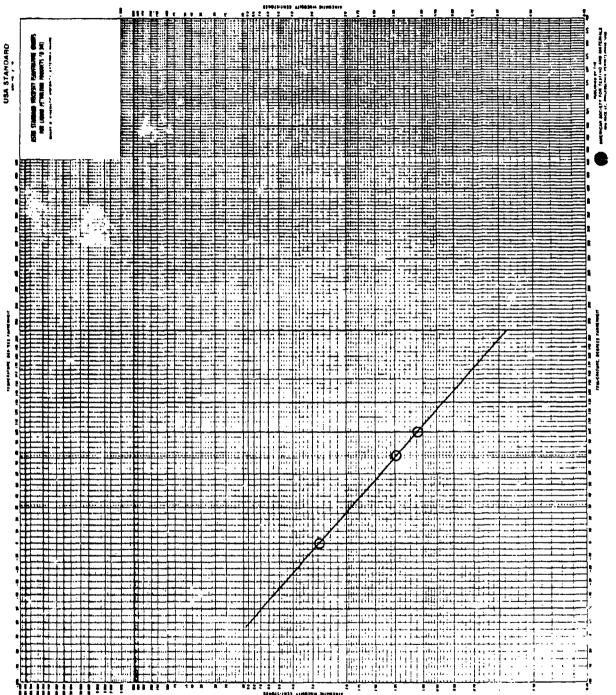
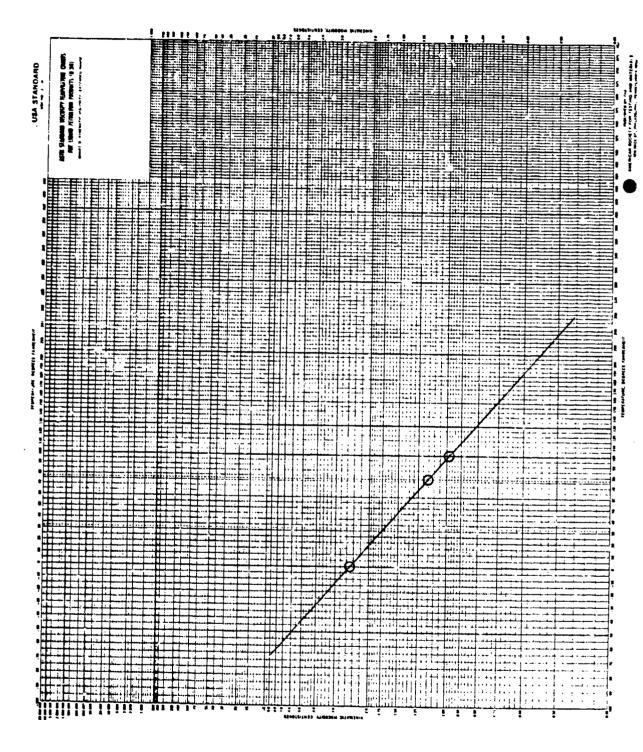


Figure 146. Viscosity/temperature plot for fuel sample from Ignition #3

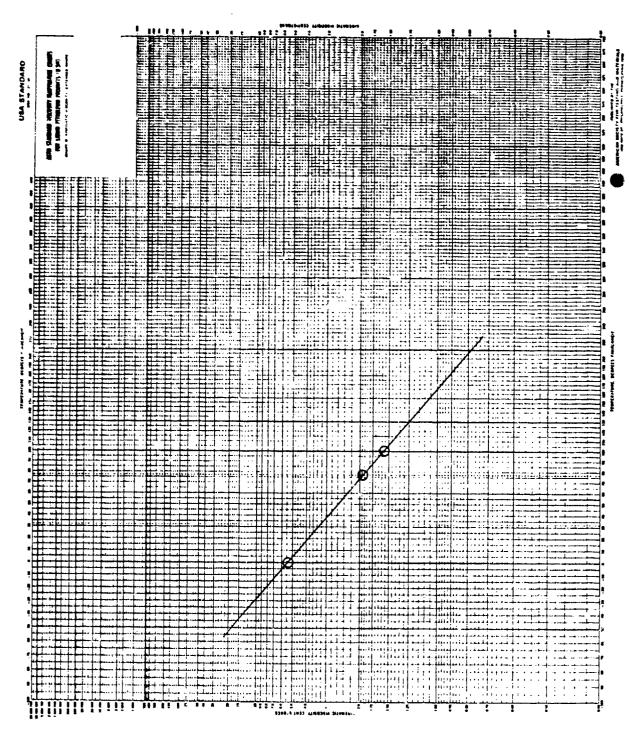


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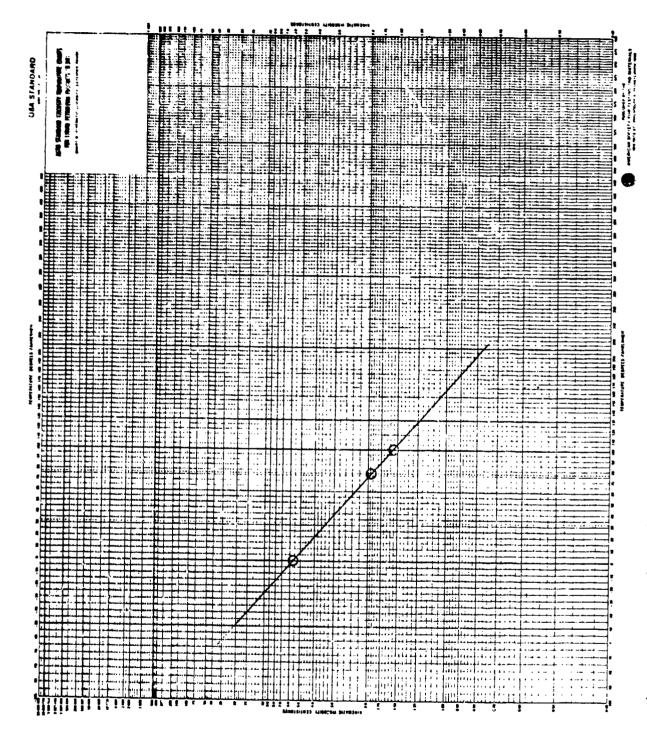
Viscosity/temperature plot for fuel sample from Ignition #5. Figure 147.



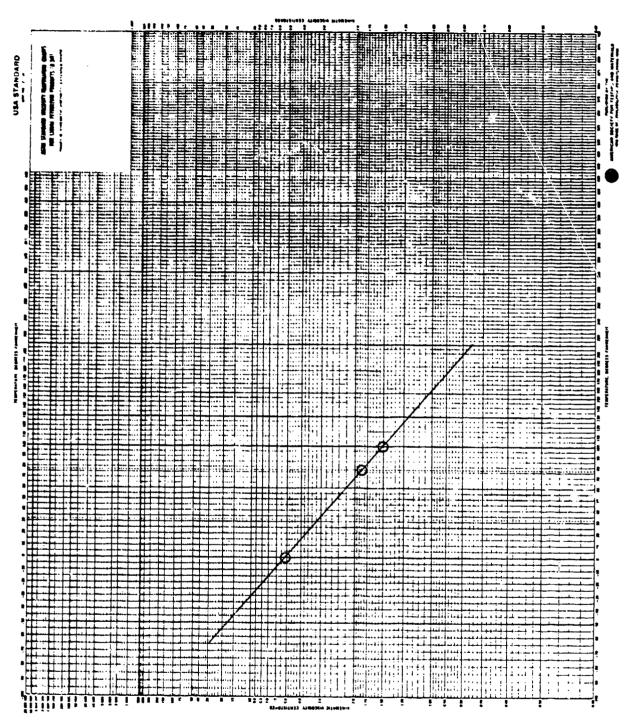
Viscosity/temperature plot for fuel sample from Ignition #6. Figure 148.



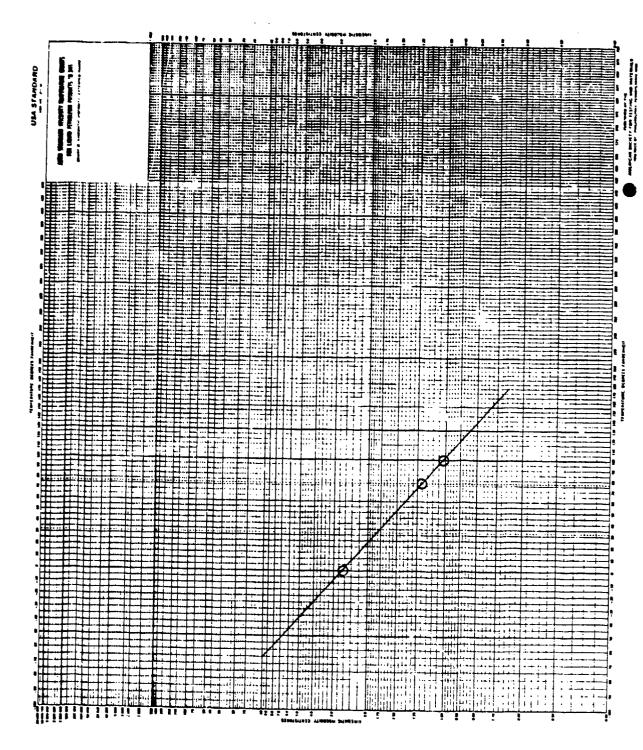
Viscosity/temperature plot for fuel sample from Ignition #7, JP-8



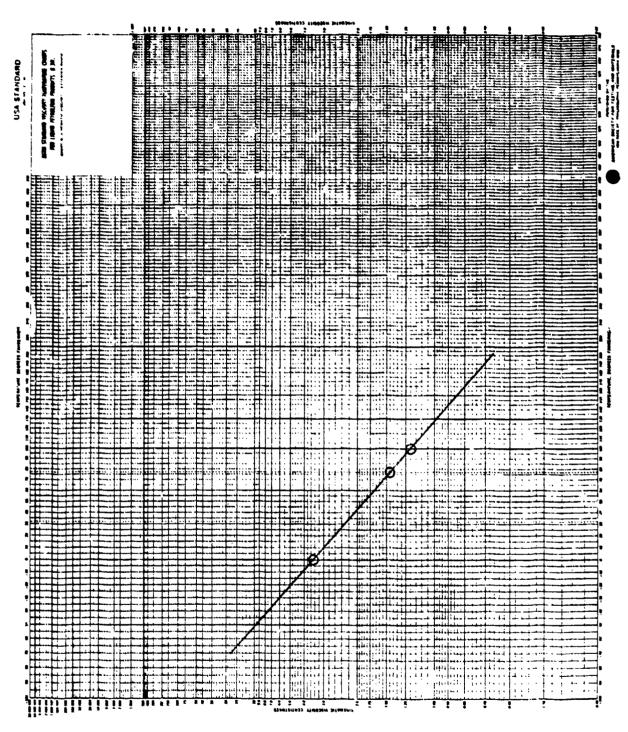
Viscosity/temperature plot for fuel sample from Ignition #8. Figure 150.



Viscosity/temperature plot for fuel sample from Ignition #9

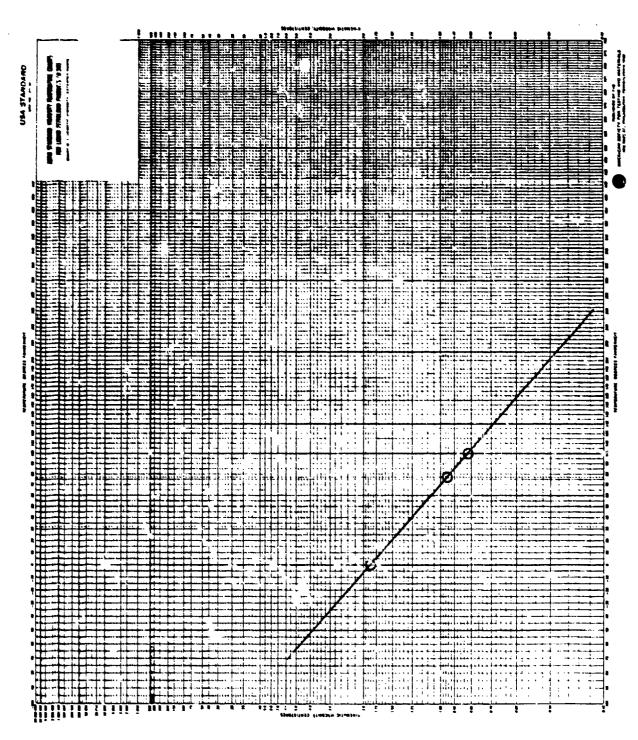


Viscosity/temperature plot for fuel sample from Ignition #10. Figure 152.



Viscosity/temperature plot for fuel sample from Ignition #11

Viscosity/temperature plot for fuel sample from Ignition #12 Figure 154.



Viscosity/temperature plot for fuel #1, Idle Point.

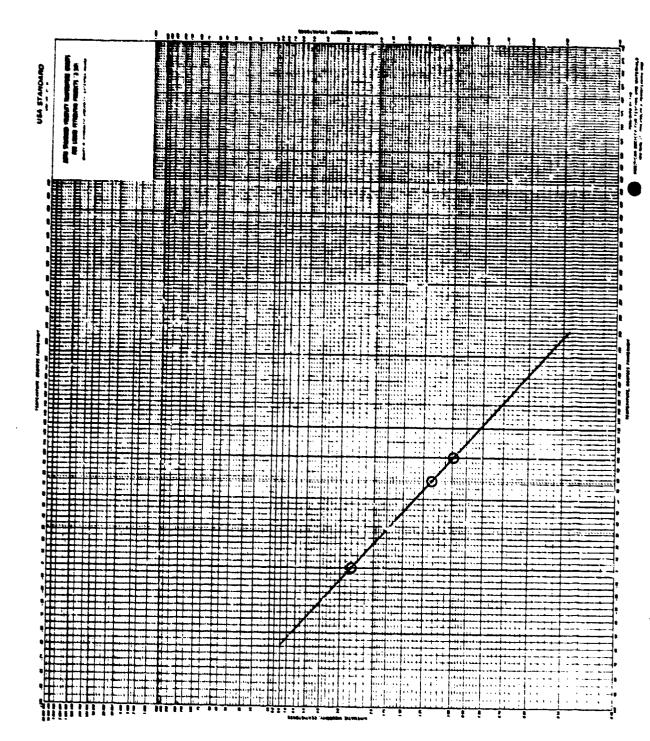


Figure 156. Viscosity/temperature plot for fuel #2.

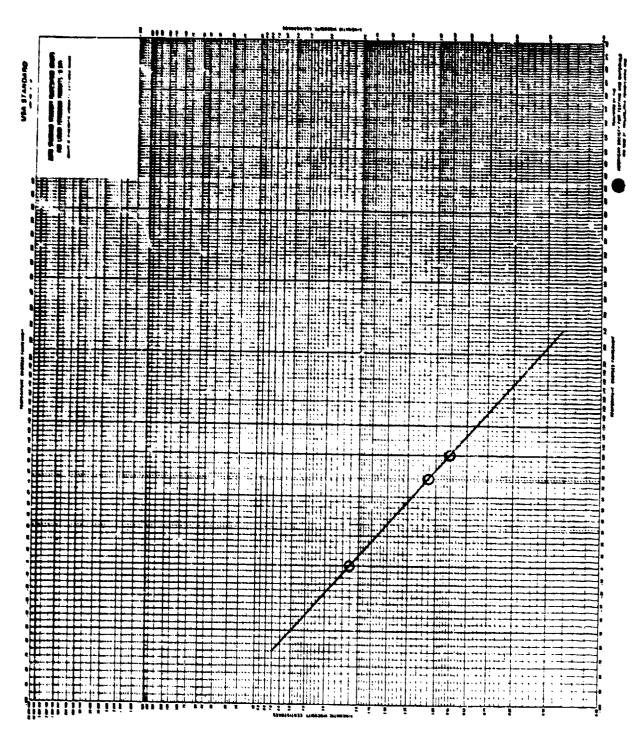
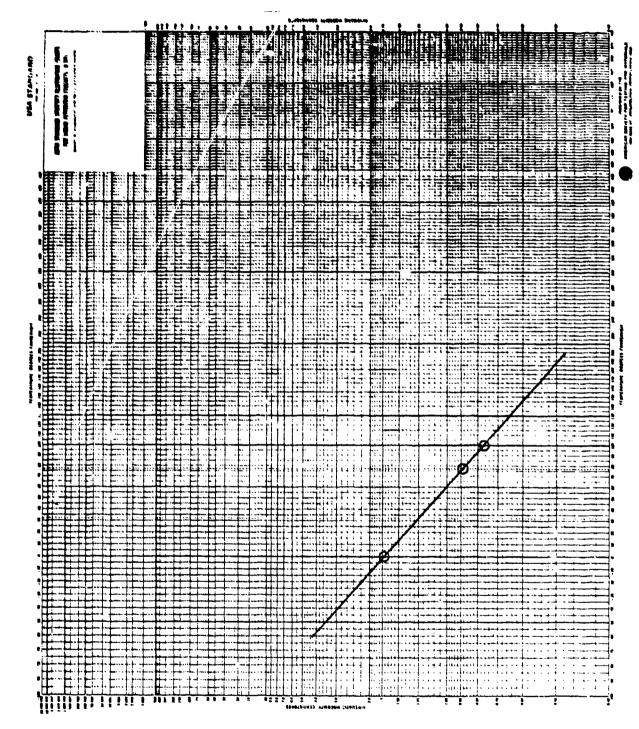
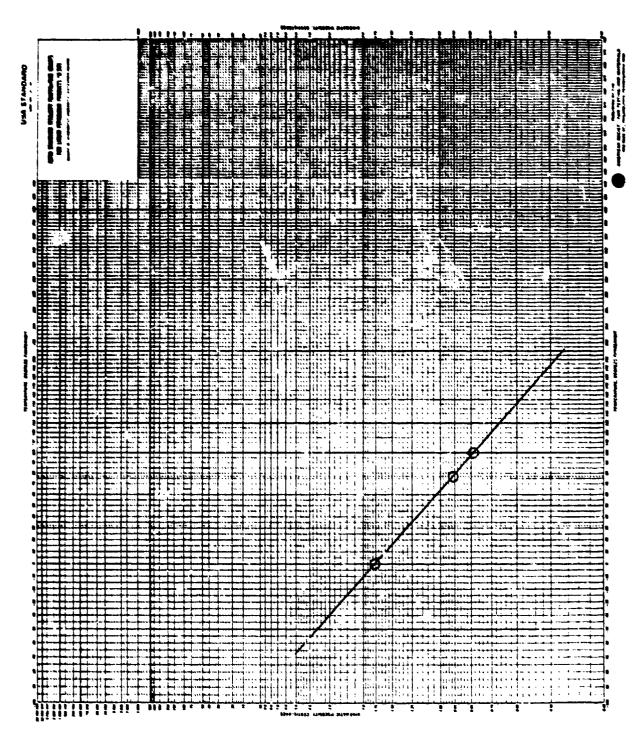


Figure 157. Viscosity/temperature plot for fuel #3.

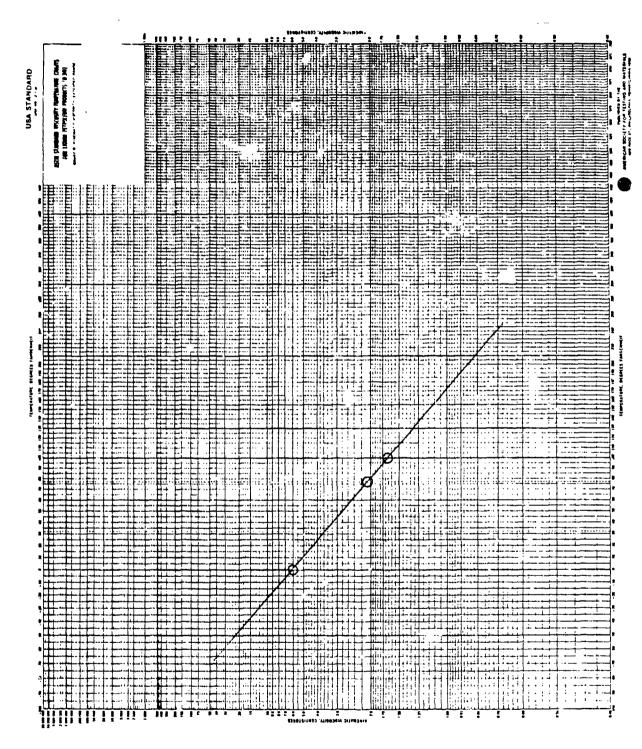


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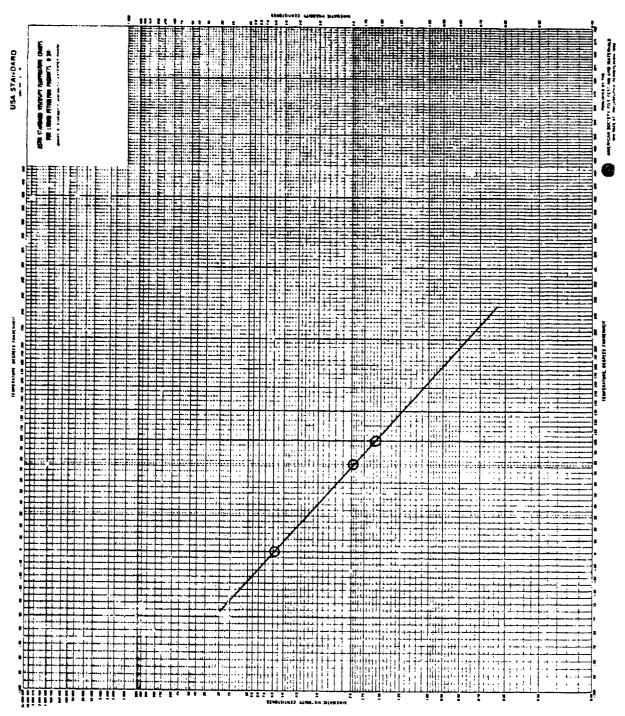
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igure 159. Viscosity/temperature plot for fuel #5.



Viscosity/temperature plot for fuel #7, Idle Point. Figure 160.



gure 161. Viscosity/temperature plot for fuel #8.

Figure 162. Viscosity/temperature plot for fuel #9.

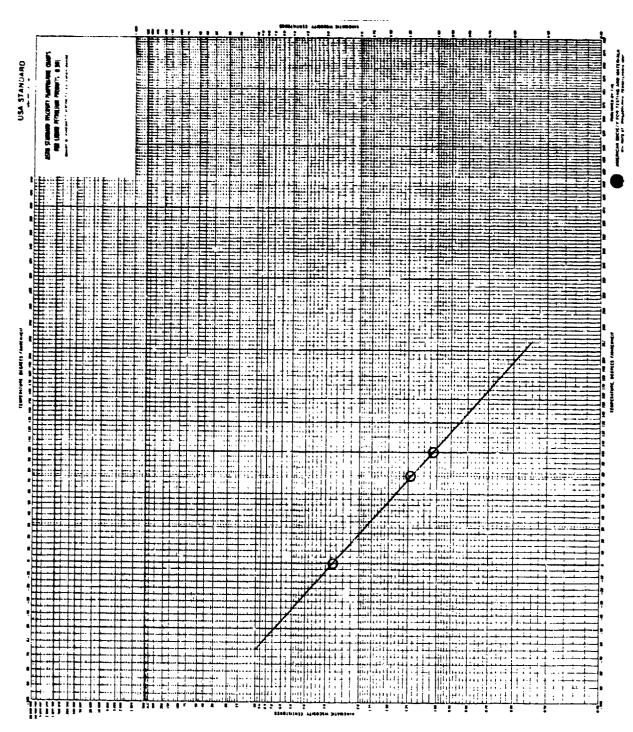


Figure 153. Viscosity/temperature plot for fuel #10.

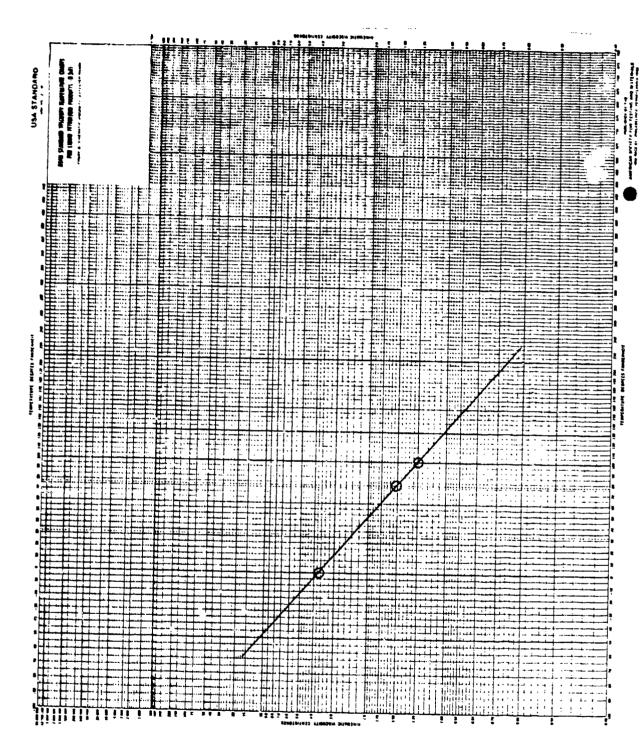


Figure 164. Viscosity/temperature plot for fuel #11.

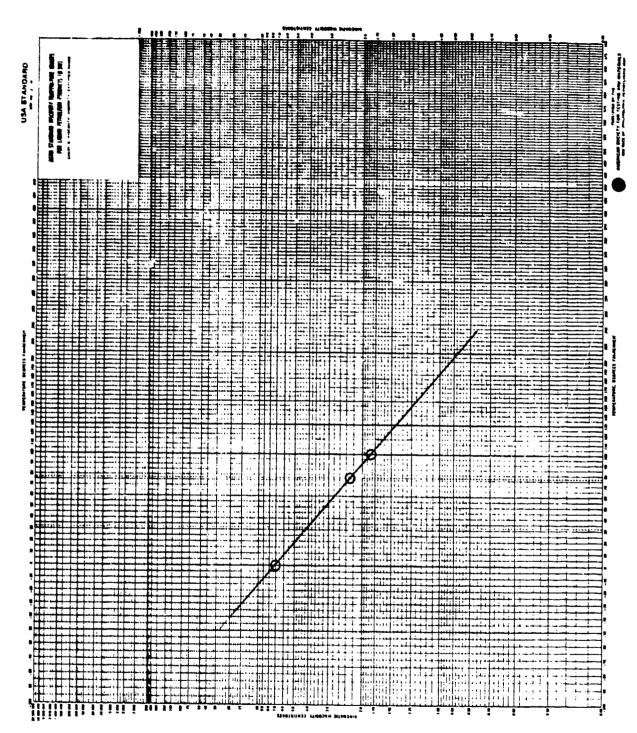


Figure 165. Viscosity/temperature plot for fuel #12.

11. CHARACTERIZATION OF DIESEL FUEL

A diesel fuel, supplied for test programs by an engine manufacturer, was characterized by analyses for hydrocarbon types and boiling range distribution. A simulated distillation by gas chromatography was conducted according to ASTM D 2887. A gas chromatograph equipped with a 3% OV-1 column and a flame ionization detector was employed for this work. Boiling range distribution data are presented in Table 160.

Hydrocarbon type analyses were performed by mass spectrometry using several different methods. The first consisted of a separation into paraffinic and aromatic fractions using the procedure described in ASTM D 2549, followed by mass spectrometric analysis using ASTM Method D 2425. This analysis was performed in duplicate. The second method used was a modification of ASTM D 2789, and was conducted at Monsanto facilities in St. Louis. The third method for hydrocarbon-type analysis was developed within Monsanto for hydrocarbon feed stocks. The boiling range limits of the method match those of the fuel quite well.

The analysis results from all three methods for hydrocarbon type distribution are shown in Table 161. All analyses are in reasonable agreement except for the distribution of the various cycloparaffins which were obtained in greater detail by the ASTM D 2549/D 2425 analysis.

TABLE 160. SIMULATED DISTILLATION OF DF-2 DIESEL FUEL (ASTM D 2887)

Perce Recove		Boiling °C	Point °F
0.5	(IBP)	127	261
1		147	297
5		179	354
10		197	387
20		222	432
30		238	460
40		255	491
50		268	514
60		282	540
70		297	567
80		313	595
90		332	630
95		349	660
99		382	720
99.5	(FBP)	391	736

TABLE 161. HYDROCARBON TYPE ANALYSIS OF DF-2 DIESEL FUEL BY THREE METHODS

AND SOUTH AND SOUTH POSSESSES TO SOUTH AND SOUTH BESTOOM BESTOOM AND SOUTH BESTOOM A

		Weig	ht percent	
	ASTM D 254	19/D 2424	ASTM D 2789	Monsanto
Comp. and type	duplicate	analyses	modified	21-PQ-38-63
	20.0	40.0	41 5	40.0
Parafí s	39.9	40.9	41.5	40.2
Cycloparaffins	19.7	19.5	34.3	35.1
Dicycloparaffins	8.2	8.0	2.3	_
Tricycloparaffins	2.5	2.5	a	-
Alkylbenzenes	9.0	8.5	8.6	10.0
Indans/Tetralins	4.7	4.6	6.5	5.3
Indens	2.6	2.7	-	1.0
Naphthalene	0.3	0.3	-	-
Naphthalenes	8.0	8.4	6.8	8.4
Acenaphthenes	2.6	2.7	-	-
Acenaphthylenes	1.6	1.8	-	-
Tricyclic Aromatics	0.9	1.0	_	-

Dash indicates that the compound type is not included in the analysis.

12. SODIUM AND WATER ANALYSES OF 2040 SOLVENT SAMPLES

Four samples of 2040 solvent blending stock were analyzed for sodium content by atomic absorption spectroscopy to assure that the salt levels were not high enough to damage fuel handling equipment during combustion testing. Three of the samples were also analyzed for water content by the Karl Fischer method. Each 2040 solvent sample represented a different step in a clean-up procedure conducted by Aero Propulsion Laboratory personnel.

<u>Sample 80036</u> was collected downstream of the clay filter tower on 12-8-80. It had been treated by a single pass through the filter separator, the NaCl salt tower, and the clay tower.

Sample 80036-a was collected from Compartment 4 of the 8400-gallon trailer after at least three passes through the filter separator, salt tower, and clay tower. No visible free water was detected in the amber liquid, but some black particles up to an inch in length (possibly hose lining or gaskets) and gravel size rust-colored particles were observed in the bottom of the tank.

Sample 80036-b was collected from the bottom-fill line of Compartment 3 of the 8400-gallon trailer. The compartment contained approximately 750 gallons of solvent when it was sampled. Solvent from Compartment 4 was passed through an anhydrous calcium sulfate drying tower, through an 18-element filter separator, and then through a 3-element clay filter before being emptied into Compartment 3, which had been carefully rinsed with methanol, water, methanol, and then clean 2040 solvent prior to filling.

Sample 80036-c was also taken from the bottom fill line of Compartment 3, but after the compartment was completely filled on 12-11-80.

Water analyses were conducted on the foregoing fuel specimens after thorough agitation to insure that both free and dissolved water were included in the determination. The sodium and water analysis results are shown in Table 162.

TABLE 162. ANALYSIS OF FOUR 2040 SOLVENT SAMPLES

Sample	Sodium ppm by wt	Water ppm by wt
80036 80036-a 80036-b	0.13 0.05 0.10	_a 175 220 ,
80036-c	0.05	150, 140 ^b

aNot determined.

13. SIMULATED DISTILLATIONS AND VISCOSITY DETERMINATIONS FOR 17 FUEL BLENDS

Samples of blends being considered for use in major engine test programs were analyzed for boiling range distribution and kinematic viscosity, to aid in determining the most suitable blends.

Simulated distillations were conducted by gas chromatography as described in ASTM Method D 2887, and data are presented in Table 163. This table and Table 164 also contain the composition of each of the seventeen fuel blends.

Kinematic viscosity was determined by ASTM Method D 445 at 77°F only. Results are presented in Table 164.

b Second analysis was conducted 24 hours later.

TABLE 163. SIMULATED DISTILLATIONS OF FUEL BLENDS

.s%																		
,AXA																		
,5%																		
. * * * * * * * * * * * * * * * * * * *																		
',·																		
		TAP	SLE 1	63.	SI	MIJI	LATEI	ות כ	STI	LLAT	'ION	S OF	· FU	EL E	LEN	DS		
		4.00	, ub 4					MOO	3-4	1400	 3- 5	MOO	3-6	M00	3-7			
	Sample No.: Blend (Vol.%)	30%	13-1 DF-2 JP-6	20%	3-2 DF-2 JP-8	10% 90%		50x 5x 45x	yl B	50% 10% X 40%	yl B	45% 53 % 50%	/1 B	40% 10% X 50%	yl B	MO0: 50% 50%	DF-2	MOO
	Percents	<u>٠</u> ٢	<u>°F</u>	<u>•c</u>	°F	<u>•c</u>	<u>•</u> F	<u>•c</u>	<u>•</u> F	<u>°С</u>	<u>•</u> F	<u>•c</u>	<u>•</u> F	<u>•c</u>	<u>•</u> F	<u>•c</u>	<u>•</u> F	<u>•c</u>
	0.5 (IBP)	138	280	137	279	136	277	47	117	48	118	41	106	43	109	46	115	133
	1.0	148	298	149	300	148	298	60	140	61	142	57	135	59	138	63	145	146
	5	176	349	174	345	174	345	91	196	93	199	89	192	90	194	97	207	173
	10	189	372	187	369	187	369	116	241	116	242	107	225	109	228	112	234	185
	20	201	394	199	390	198	388	148	298	152	306	140	284	141	286 324	148	298	197
	30	213	415	211	412	210	410 424	173 198	343 388	168 195	334 383	165 192	329 378	162 179	354	185 808	361 406	208 216
	40 50	222 232	432 450	219 229	426 444	218	439	218	424	216	421	213	415	204	399	556	439	223
	60	240		236	457	236		236	457	234	454	231	448	225	437	243	4.19	232
	70	253	487	249	480			254	489	252	486	249	480	244	471	260	500	240
	80	265	509	259	498	255	491	274	525	273	523	269	516	265	509	281	538	251
	90	285	545	278	532	270	518	300	572	301	574	299	570	295	563	308	586	264
	95	309	503	299	570	285	545	321	610	320	608	317	603	316	601	326	619	272
	99	353	667	344	651	329	624	344	651	359	678	358	678	356	673	362	684	292
	99.5 (FBP)	369	696	360	680	347	657	372	702	37 i	699	371	700	370	698	373	703	30
	Sample No.: Blend (Vol.%)	90	1003-10 17 JP-8	851	3-11 JI-8	801	3-12 (JP-8 (Xy) B	M003 95% 5%		90%	3-14 JP-4 Xyl B	85%	3-15 JP-4 Xyl B		3-16 -4	MOO3 Tynd Sha JP	la I I	
	Percents	<u>°c</u>	°F	<u>•c</u>	<u>•</u> F	<u>•c</u>	<u>•</u> F	<u>•c</u>	°F	<u>°C</u>	<u>°F</u>	<u>•c</u>	°F	<u>•c</u>	<u>• F</u>	<u>•c</u>	•F	
	0.5 (IBP)	136	277	136		138	280	31	88	31	87	34	93	31	88	151	304	
	1.0	144	291	142		143		37	98	37	98	40	105	38	101	155	310	
!	5	160	320	159	318	158	316	68	154	68	154	71	160	68	155	164	328	
, 	10	166	331	162	324	163		89	192	89	191	91	196	87	189	170	338	
ļ	20	187			349		338	106	222	108	226	115	239	102	215	178	352	
	30	199			383		374	120	249	125	257	127	261	118	245	184	369	
1	40		412		404	203		141	286	143	289	147	296	135	274	197	386	
	50		426		421		420	160	320 345	161 169	322 336	162 169	323 336	153 175	306 347	203 213		
	60 70	228			432 455		435 455	174 196	345 385	190	375	187	358	175	384	219	427	
	70 80	236 249			433 477		433 477	216	421	212		210	410	216	421	230	445	
	90	261	502		500	261		236	458	234	454	234	453	235	455	240		
	95	271	520		518	271		252		250	482	249	481	250	483	252		
	39	291	556	289		290		274	525	265	509	273		271	520	275		
	99.5 (FBP)	301			567		568		545		545		544	202	539	***	546	

THE PRODUCTION OF THE PROPERTY

TABLE 164. KINEMATIC VISCOSITY OF FUEL BLENDS AT 77°F

			positions		
Sample		in volum	e percen	<u>t</u>	Viscosity,
number	JP-4	JP-8	DF-2	Xyl B	centistokes
M003-1		70	30		2.298
M003-2		80	20		2.220
M003-3		90	10		2.146
M003-4	45		50	5	1.526
M003-5	40		50	10	1.514
M003-6	50		4 5	5	1.440
M003-7	50		40	10	1.353
M003-8	50		50	 -	1.544
M003-9		100			2.064
M003-10		90		10	1.821
M003-11		85		15	1.727
M003-12		80		20	1.626
M003-13	95			5	0.9188
M003-14	90			10	0.9118
M003-15	85			15	0.9032
M003-16	100				0.9284
M003-17	- · ·	100			1.594
	r	yndall Shale JP	_ 8		_,

14. SIMULATED DISTILLATIONS, VISCOSITY DETERMINATIONS, AND HYDROCARBON-TYPE ANALYSES (ONE SAMPLE ONLY) FOR 19 AEDC FUEL BLENDS

Simulated distillation analyses and kinematic viscosity determinations at 70°F and 32°F were conducted on the 19 fuel samples.

One sample was also analyzed for hydrocarbon type by mass spectrometry. These analyses were conducted to aid in determining the most suitable blends for use in a major engine test program to be conducted at Arnold Engineering and Development Center (AEDC).

The simulated distillation analyses were conducted according to ASTM Nethod D 2887. The results are presented in Table 165. This table also contains results for selected repeat analyses.

Kinematic viscosity was determined at 32°F and 72°F by ASTM Method D 445. The results are presented in Table 166.

TABLE 165. SIMULATED DISTILLATION OF AEDC FUEL BLENDS

	ا - اص				
	006-5A	277 288 314 326 336 339 342 422 441 457 480 506 506			
	<u> </u>	136 142 157 163 170 189 204 217 227 227 236 749 262 274 3302			
	4004 C	292 321 321 339 421 440 440 466 550 551 543 559	إحراب	304 328 328 401 445 491 568 568 615 615 700	: 9
		144 1160 1160 1160 1160 1160 1160 1160 1	7. 5006-7	151 3 165 3	•
	O'A I				, 5
Š	00 0 -30	139 157 219 265 265 358 410 477 477 499 528 664 664 669	49-900H	282 303 352 378 407 476 476 493 476 493 476 476 476 476 476 476	267
BLENDS	왕이	60 69 104 129 181 232 247 260 276 295 313 313	9	139 150 178 192 222 234 247 256 256 269 308	382
_	워터	124 145 205 205 2245 334 334 416 417 477 477 503 533 533	احاق		. ~
LOEL	J. 2008-3C	51 1 63 1 96 2 1118 2 118 39-900V	8 281 0 301 0 301 1 430 1 453 4 468 6 474 5 490 5 490 5 490 6 543 6 692 6 692		
7			XP (138 150 170 197 209 21 214 214 214 214 214 214 214 214 214	38
AEUC.	006-38	124 145 216 216 256 347 431 457 485 510 540 580 616 685	09-900 0	283 304 352 378 378 407 429 451 473 492 514 553	722
2	<u>ğ</u>	51 63 99 1125 1175 202 202 203 205 305 305 313	0	140 151 178 192 208 220 233 245 255 255 255 308	
5	45	103 135 136 240 240 304 304 412 412 444 472 530 530 510 511	Ulad		
Ę	490CH	39 10 57 11: 57 11: 51 11: 51 11: 52 11: 52 11: 52 44: 52 44: 52 44: 52 44: 52 54: 53 54: 54: 56: 57: 58: 58: 58: 58: 58: 58: 58: 58: 58: 58	29-900M	1 309 1 327 363 1 413 4 413 4 454 4 85 5 85 5 85 5 74 5 91 6 61 7 706	•
1		3 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	361	154 164 195 195 212 234 253 253 263 276 288 301 310 315 375	393
	Repeat 1006-20 0 F	93 104 166 198 242 242 280 305 373 378 421 421 421 456 578	\$ ₽	277 300 350 377 405 449 449 510 510 516 616	22
-		34 40 40 75 1117 1117 1152 1162 1175 1192 216 2315 287	#9-900M	136 1149 1177 1191 220 222 223 24 265 303 303 303 305 67 665	
3	Q[N]	L M W W L M W L M W W M M M M M M M M M	and to the		
•	27-900 2009-30	97 9 111 9 172 9 203 7 206 7 206	Repeat HO06-6A	312 335 382 408 435 481 499 523 544 572 722	•
	X P_[36 44 78 78 97 117 113 115 116 117 117 119 119 218 218 218 218 218 218 218 218 218 218	등 되다	156 168 194 209 224 249 259 273 264 373 384 386 386	395
	Pepear:	93 105 105 120 206 246 289 337 337 459 465 588	5 -	297 318 318 318 318 318 318 421 437 443 475 541 541 628	œ.
	9 0 c	34 76 97 1119 1169 1169 1169 221 221 221 221	49-900H	1147 2 11	•
	mi s.i				\$
,	006-28 C F	97 109 109 109 204 248 248 316 316 316 316 420 420 485 582	2006- 20- 30- 30- 30- 30- 30- 30- 30- 30- 30- 3	296 321 391 414 413 443 443 443 443 443 443 443 543 543 54	297
	장이	36 43 76 96 96 1118 1142 1159 1163 1163 1163 1163 1163 1163 1163 116	ğirl	146 160 1187 1199 212 222 222 224 246 255 265 278 287	114
	র •।	86 98 155 155 232 232 258 320 320 347 442 472 472 553	2 A B	290 294 315 310 315 315 316 425 447 447 469 469	5
	M006-2A	30 69 112 90 112 112 112 114 114 114 114 114	Repeat M006-5s		
			- 4.5	143 117 117 117 116 1186 1186 203 203 218 225 273 273 273	3
		57 98 152 162 210 243 266 298 337 413 413 526 526	M006-58	351 356 335 337 3391 339 454 468 513 513 573	
	<u> </u>	14 37 67 63 93 1117 1130 1148 1162 212 2212 2247	<u> </u>	177 180 191 191 197 203 209 214 253 253 253 304 319	3
	Sample: Scale: nts	•	le:		-
	Sample: Temp. Scale: Percents	0.5(18P) 1.0 5.0 10 20 20 30 40 60 60 60 60 60 60 60 70 70 70 80 80 80 80 80 80 80 80 80 80 80 80 80	Sample: Temp, Scale: Percents	5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0	
	E S	0.5 5.0 10.0 10.0 10.0 10.0 10.0 10.0 10	San Temp, Sc Percents	0.5(IBP 1.0 5.0 110 30 30 40 40 40 60 60 90 99 995	

TABLE 166. KINEMATIC VISCOSITY OF AEDC FUEL BLENDS

Sample	Centistokes					
number	32°F	70°F				
M005-1	1.271	0.9493				
M006-2A	1.223	0.9204				
M006-2E	1.309	0.3689				
M006-2C	1.267	0.9455				
M006-3A	2.844	1.870				
M006-3B	3.402	2.143				
M006-3C	3.039	1.964				
M006-3D	3.660	2.291				
M006-4	3.524	2.223				
M006-5A	2.572	1.710				
M006-5B	2.161	1.485				
M006-5C	3.407	2.130				
M006-6A	4.657	2.765				
M006-6B	4.795	2.832				
MC06-6C	7.610	4.091				
M006-6D	4.895	2.888				
M006-6E	5.013	2.934				
M006-6F	5.209	3.045				
M006-7	10.75	5.361, 5.369				
Mixture ^a	3.715	2.330				

aMixture of 3 parts Sample M006-7
with 2 parts M006-1.

Mass spectrometric hydrocarbon-type analyses were conducted for sample M006-7 using both a modification of ASTM D 2789 and Monsanto Method 21-PQ-38-63. Results are given in Table 167.

TABLE 167. HYDROCARBON-TYPE ANALYSIS OF AEDC FUEL BLEND M006-7

Components	ASTM D 2789 volume %	Monsanto 21-PQ-38-63 weight %
Paraffins	46. 0	43.0
Monocycloparaffins	33.9	39.9
Dicycloparaffins	2.0	-
Alkylbenzenes	5.8	7.0
Indans/tetralins	3.5	3.4
Naphthalenes	2.2	5 .5
Indenes/dihydronaphthalenes	-	1.2

15. PARTIAL CHARACTERIZATION OF SIX AEDC BLENDING STOCKS

Kinematic viscosity, simulated distillations, and hydrocarbon-type analyses were conducted on six JP-4 and JP-5 fuels blended with two aromatic stocks designated as xylene bottoms and A-400. These fuels were to serve as blending stocks for use in preparing other fuels for test programs at AEDC.

Kinematic viscosity was determined by ASTM Method D 445 at 70°F only. Results are presented in Table 168.

TABLE 168. KINEMATIC VISCOSITY AT 70°F

		Sample des	scription		
Number	Percent JP-4	Percent JP-5	Percent Xy-B	Percent A-400	Centistokes
WEH-81-5	82.7		11.6	5.7	0.9605
WEH-81-6	82.5		12.0	5.5	0.9708
WEH-81-7	82.0		13.0	5.0	0.9588
WEH-81-8		69.5	24.5	6.0	1.659
WEH-81-9		69.0	25.5	5.5	1.643
WEH-81-10		69.0	26.0	5.0	1.637

Simulated distillations were conducted by gas chromatography as described in ASTM Method D 2887. Data are presented in Table 169.

Hydrocarbon-type analyses were conducted by mass spectrometry using modified ASTM Method D 2789. Data are presented in Table 170.

16. PARTIAL CHARACTERIZATION OF 24 COMBUSTION TEST FUELS WITH VN NUMBERS

Samples of fuels from engine test programs were sufficiently characterized to correlate fuel composition and properties with fuel performance.

TABLE 169. SIMULATED DISTILLATION OF FUEL BLENDS

		81-5		81-6		81-7		81-8		81-9		61-10
	_	JP-4		JP-4		JP-4		JP-5		JP-5		JP-5
Percent		Xy-B A-400		Ху-В A-400		Xy-B A-400		-		ХУ-В A-400		ху-в A-400
Recovery	<u>°C</u>	6 <u>F</u>	<u>°C</u>	°F	°C	°F	<u>°C</u>	F	°C	°F	°C	°F
	_						_					
0.5(IBP)	35	94	40	103	37	98	141	286	141	286	142	287
1	53	128	57	134	56	132	146	295	145	294	146	295
5	72	162	76	168	73	163	154	309	152	306	152	306
10	88	191	89	191	98	191	158	316	158	316	158	316
20	112	234	113	235	113	236	164	328	163	326	163	326
30	127	261	130	267	130	266	184	363	181	358	180	357
40	150	303	152	306	154	309	197	387	196	385	196	384
50	162	324	162	324	162	323	208	407	207	405	207	405
60	185	365	186	366	184	363	219	425	218	424	218	424
70	205	400	205	4C1	205	400	230	445	229	444	229	445
80	225	438	225	437	224	435	243	47 0	243	469	243	469
90	245	474	247	476	246	474	259	498	258	497	258	497
95 .	260	501	261	502	261	502	272	522	272	521	271	520
99	296	564	299	571	297	566	306	583	303	577	301	574
99.5(FBP)	312	593	315	599	312	593	320	608	312	594	311	591

TABLE 170. HYDROCARBON-TYPE ANALYSES BY ASTM D 2789 (MODIFIED)

Components	WEH-81-5 82.7 JP-4 11.6 Xy-B 5.7 A-400	WEH-81-5 82.5 JP-4 12.0 Xy-B 5.5 A-400	WEH-81-7 82.0 JP-4 13.0 Xy-B 5.0 A-400	WEH-81-8 69.5 JP-5 24.5 Xy-B 6.0 A-4	WEH-81-9 69.0 JP-5 25.5 Xy-B 5.5 A-400	WEH-81-10 69.0 JP-5 26.0 Xy-B 5.0 A-400
Paraffins	54.6	54.5	54.1	32.4	32.0	31.9
Monocyclo- paraffins	16.1	16.1	16.0	24.5	23.9	24.2
Dicyclo- paraffins	2.9	2.9	2.9	2.5	2.6	2.4
Alkyl benzenes	20.7	21.1	21.7	31.2	32.5	32.8
Indans & Tetralins	2.6	2.5	2.5	4.8	4.6	4.5
Naphthalenes	3.1	2.9	2.8	4.6	4.4	4.2

Density was determined at 32°F, 70°F, and 100°F by the dilatometer method. Data are presented in Table 171.

TABLE 171. DENSITY AS A FUNCTION OF TEMPERATURE

	Grame ner	cubic cen	timeter
Sample	32°F	70°F	100°F
Dampie			100 1
VN-81-84	0.7739	0.7575	0.7441
- 85	0.8197	0.8044	0.7915
-86	0.8213	ე.8059	0.7937
-87	0.8708	0.8552	0.8427
-88	0.8337	0.8182	0.8053
-89	0.8487	0.8324	0.8195
-90	0.8458	0.8298	0.8172
~91	0.8505	0.8342	0.8212
- 92	0.8179	0.8607	0.7879
~93	0.8307	0.8130	0.7999
-94	0.8075	0.7910	0.7774
- 95	0.7905	0.7740	0.7610
- 96	0.8561	0.8413	0.8294
- 97	0.7743	0.7575	0.7448
-98	0.8489	0.8324	0.8192
- 99	0.8181	0.8019	0.7884
-100	0.8327	0.8151	0.8018
-101	0.8081	0.7914	0.7780
-102	0.7926	0.7763	0.7631
-103	0.8254	0.8104	0.7978
-104	0.8763	0.8602	0.8482
-105	0.8497	0.8339	0.8217
- 106	0.8518	0.8353	0.8225
-107	0.8402	0.8237	0.8113

Naphthalenes content was determined by ASTM D 1840. This method covers the determination by ultraviolet spectrometry of the total concentration of naphthalene, acenaphthene, and alkylated derivatives of these hydrocarbons in straight-run jet fuels containing not more than 5 percent of such components and having end points below 315°C. The method determines the maximum amount of naphthalenes that could be present by measurement of the absorbance at 285 nm of a solution with a known concentration of fuel. The results are presented in Table 172 where they are compared against naphthalenes content determined by the ASTM D 2789 hydrocarbon-type analysis. The results by the UV absorbance method are obviously

TABLE 172. NAPHTHALENES BY ASTM D 1840 METHOD

Sample	D 1840 weight %	Comparative D 2789 analysis, volume %
VN-81-84	6.3	0.7
-85	10.8	1.8
-86	10.3	1.7
-87	106, 110	22.9
-88	8.0	1.4
- 89	5.8	0.9
- 90	63.7	12.5
-91	124	25.1
-92	78.3	14.7
- 93	3.0	0.4
-94	4.3	0.5
- 95	5.7	0.6
- 96	52.9	6.0
- 97	6.2	0.7
-98	116	25.0
- 99	81.7	14.8
-100	3.0	0.4
-101	4.2	0.5
-102	5.4	0.6
-103	10.6	2.1
-104	116	23.2
-105	60.6	12.3
-106	2.8	1.0
-107	4.1	1.5

not meaningful as they are three to nine times higher than those from the hydrocarbon-type analysis, and three values are actually greater than 100%.

Simulated distillations were conducted by gas chromatography as described in ASTM Method D 2887. As requested because of the large number of samples, the GC runs were conducted in an automated fashion with some of the customary individual handling of data being eliminated. The percent cumulative area counts are reported directly as recorded by the data system. Results are presented in Table 173.

SIMULATED DISTILLATIONS BY GAS CHROMATCGRAPHY TABLE 173.

VN-81-106	266 312 312 312 313 313 313 313 421 421 421 421 421 421 421 421 421 421		VN-81-107	279	5 60	324	347	9	453	475	504 529	268	290
<u>\$</u> 0	130 138 150 155 162 164 164 172 172 195 216 233 255 270	308	충이	137	154	162	175	210	234	246	262	298	310
VN-31-94	84 100 100 100 205 205 282 282 293 311 311 442	529 550	VN-81-105	566	345	363	410	45	4 69	493	5 26	579	665
\$ 5	29 88 81 81 86 86 1130 1145 1155 1155 1156 1156 1156 1156	276 286	- NS	139	Į.	8 8	210	228	243	256	269 279	304	315
3. J.	97 131 130 234 286 288 288 320 320 332 415	\$20 \$47	VN-81-104	259	1	370	412	450	469	493	518 538	585	601
* L	36 55 68 88 112 114 114 116 116 116 117 117	2.17	\$ 0 8	126	175	188	211	232	243	256	27.5 281	307	316
VA-31-92	91 106 1183 202 202 291 354 403 482 482 482	613	VN-91-103	271	342	363 388	406	435	468	169	532	574	594
\$ 0	33 64 176 176 179 205 223 224 226 226	323	\$ 0 S 0	133	172	184 198	208 218	224	242	255	3.7 2.76	30).	315
VN-E1-91	122 232 253 257 361 469 577 578 578 578	649	VN-81-102	8 2	165	194 241	306	333	414	459	237 292	646	662
\$ 0	50 61 111 111 111 115 115 115 116 116 116	343	8 0	30	7.	90 116	130	167	212	237	311	341	350
06-18-NA	291 318 353 405 433 455 433 455 554 558 610	658	161-18-NA	98 109	178	25. 24.4	289 318	325	345	390	482	535	554
8 v	144 159 195 207 223 247 247 257 257 291 308	348	- S	ŏ 1	18 3	11 % 11 %	143 159	163 167	174	199	250	278	290
9-18-NA	261 277 282 284 208 336 336 340 361 361 491 523	594	VN-81.100	100	192	286 286	30 9	329	3.40	352	469	541	568
8-NO:	137 136 139 140 140 162 165 171 183 202 226 225 225 225	312	<u>\$</u>	38	68	14.	154 163	165 167	171	178	243	283	298
VN-81-68	248 270 284 293 324 336 336 34 4-1 4-1 4-1 538	595	6-1-3	95	162	741 741	262 313	363 403	433	455	507	563	286
NN 0	120 132 140 145 162 169 181 197 212 227 227 243 266 281	313	4. D.	28 35	27 9	116	128 156	184 200	223	235	264	295	308
VN-81-87	243 326 324 345 345 347 417 442 442 455 550	617	-188	96 106	180	246	302 361	399 428	453	600	523	579	593
<u>\$</u>	117 130 162 174 174 190 203 228 228 241 241 257 275 275	325	VN-81-98	8 4	82	119	150 183	20 4	234	258	273	304	CTE
VN-81-65	246 264 316 336 336 333 452 485 518 563 646	999	6 3	98	189	277	243 266	302 347	387	462	491	531	20
N.	119 129 158 169 169 111 223 223 223 223 223 237 253 253 270 253 312	352	VN-81-97	30 30	% E	01	130	150 175	197	239	255	277	007
3. D.	237 257 313 313 381 395 442 442 464 464 691 523	597	S 1-1	255 286	349 381	421	480	50 4 531	561	642	676	770	611
N C	114 125 156 167 167 194 204 217 228 240 255 273 286 309	314	VN-81-95	1.24 141	176 194	216	249	262 271	294	339	358	398 415	;
VN-81-84	79 84 1151 1187 2212 224 224 345 345 424 424 462 462 462	531	S 1-1	85 95	160 192	239	289	324	406	529	230	660	700
- NA	26 29 66 86 100 117 127 127 174 174 197 239 239 239	71.2	4N-81-95	35.0	17 68	33	143	162	208	276	310	3.0	}
Percent. Recovered	0.5 (IBP) 1 5 2 20 2 20 3 3 3 3 5 5 6 6 6 6 6 6 6 6 6 6 6 6 6 6	99.5(FBP)	Recovered	0.5 (IBP)	. 0 <u>.</u>	20	2 9 5	2 9 6	0 00	06	95	99.5(FB2)	

Hydrocarbon-type analyses were conducted by mass spectrometry using a modification of ASTM D 2789. Data are presented in Table 174. Twelve of the samples were also analyzed by the Monsanto mass spectral method for hydrocarbon feed stocks, and these data are presented in Table 175, along with comparative data from the ASTM method.

TABLE 174. HYDROCARBON-TYPE ANALYSES BY ASTM D 2789 (MODIFIED)

			Volume	Percents		
Sample	Paraffins	Monocyclo- paraffins	Dicyclo- paraffins	Alkyl- benzenes	Indans & Tetralins	Naphtha- lenes
VN-81-84	63.4	21.3	5.2	8.1	1.3	0.7
85	45.3	40.0	2.0	7.8	3.1	1.8
86	46.5	39.8	1.6	7.4	3.0	1.7
87	31.3	27.5	1.3	11.6	5.4	22.9
88	36.0	31.3	1.8	28.7	0.8	1.4
89	25.9	21.3	1.6	50.3	0.0	0.9
90	38.1	33.7	1.7	9.7	4.3	12.5
91	37.5	20.1	0.0	12.7	4.6	25.1
92	46.2	24.9	0.0	11.0	3.2	14.7
93	32.4	10.7	2.6	53.8	0.1	0.4
94	45.2	14.9	3.6	35.2	0.6	0.5
95	58.0	20.1	5.0	15.2	1.1	0.6
96	43.0	33.9	2.3	8.5	6.3	6.0
97	62.9	21.6	5.3	8.2	1.3	0.7
98	37.6	20.2	0.0	12.6	4.6	25.0
99	46.1	24.8	0.0	11.1	3.2	14.8
100	31.5	10.8	2.5	54.0	0.7	0.4
101	45.3	15.4	3.7	34.1	1.0	0.5
102	57.6	20.2	5.1	15.0	1.5	0.6
103	42.7	40.3	3.3	7.5	4.1	2.1
104	29.5	27.6	2.2	11.4	6.1	23.2
105	36.2	34.2	2.8	9.4	5.1	12.3
106	24.4	21.4	2.0	51.2	0.0	1.0
107	33.7	31.1	2.7	29.2	1.8	1.5

17. CHARACTERIZATION OF 131 VARIOUS FUEL SAMPLES BETWEEN MAY 1980 AND JANUARY 1982

Twenty-nine groups of fuel samples comprising a total of 131 fuels were characterized either in detail or in part from May 1980 to January 1982 to obtain data that would allow correlation of physical and chemical properties with combustion performance. Most

COMPARATIVE HYDROCARBON-TYPE DATA FROM TWO ANALYTICAL METHODS, IN WEIGHT PERCENTS TABLE 175.

開放を放送しているのでは、現代を含める。

	VN-81-85	-85	VN-81-86	-86	VN-81-87	-87	VN-81-88	-88	VN-81-89	-89	VN-81-90	-90
Components	D 2789 ^a	HONS	D 2789	MONS	D 2789	MONS	D 2789	HONS	D 2789	MONS	D 2785	HONS
	•	;	;	,		ç	,	•		•	7 70	2 66
Parattins	47.8		. t.	43.6	7.77	6.07	7.00	7.00	6.03	1.03		2
Cycloparaffins	42.8	42.5	42.2	41.7	27.5	26.7	33.1	31.1	22.4	20.6	34.9	34.2
Alkylbenzenes	8.5	10.1	8.1	9.5	11.9	14.8	30.9	34.6	53.0	56.8	10.3	12.6
Indans/Tetralins	3.6	3.2	3.5	3.1	5.9	4.7	6.0	1.7	0.0	0.3	4.9	4.0
Indenes	ı	0.0	ı	0.1	1	0.5	ı	0.0	ı	0.0	•	0.2
Naphthalenes	2.3	5.6	2.2	2.4	27.0	27.0	1.7	2.2	1.1	1.9	15.2	15.4
	VN-81-96	96-	VN-81-103	-103	VN-81-104	-104	VN-81-105	-105	VN-81-106	-106	VN-81-107	-107
Components	D 2789 ^a	MONS	D 2789	MONS	D 2789	MONS	D 2789	MONS	D 2789	MONS	D 2789	MONS
								,		; ;	!	,
Paraffins	39.9	39.5	40.1	39.6	26.0	24.9	33.0	32.2	22.1	19.1	31.1	28.6
Cycloparaffins	36.2	34.5	44.3	43.9	28.4	27.6	36.4	35.8	22.9	20.8	33.7	31.7
Alkylbenzenes	9.53	10.7	8.2	9.7	11.7	14.5	6.6	12.2	53.8	57.2	31.3	35.0
Indans/Tetralins	7.3	5.9	4.8	4.1	9.9	5.2	5.8	4.6	0.0	1.0	2.1	2.4
Indenes	1	9.0	ı	0.0	ı	9.0	ı	0.2	1	0.0	1	0.0
Naphthalenes	7.4	8.9	5.6	2.7	27.3	27.2	14.9	15.0	1.2	1.9	1.8	2.3
•												

ASTM Method D 2789 provides data in volume percents. The weight percent data were calculated from average density values for the compound classes.

b. Monsanto mass spectral method developed for hydrocarbon feed stocks.

Calso includes dihydronaphthalenes.

of the fuel samples had been combustion-tested at either General Electric Company, Pratt and Whitney Company, or AEDC. Generally, however, the only information submitted with the samples was a sample identification. For the sake of conciseness, data for these twenty-nine groups of samples have been consolidated in this single subsection.

Density was determined as a function of temperature for 67 samples, and these data are presented in Table 176. Kinematic viscosity was determined for 55 samples as a function of temperature, and these data are presented in Table 177. The viscosity/ temperature relationships are graphed in Figures 166 through 219. Surface tension versus temperature was also determined for 55 samples as presented in Table 178. Vapor pressure versus temperature was determined for 49 of the samples, and results are provided in Table 179. Heat of combustion was also determined for 49 samples and these data are presented in Table 180. Boiling range distribution was determined for 91 samples by GC simulated distillation, and these results are provided in Tables 181 and 182. Table 181 provides results in degrees Celsius while Table 182 provides the same realts in degrees Fahrenheit. Hydrocarbon-type analyses by modified ASTM Method D 2789 were conducted on 102 of the samples, and these data, in volume percent, are provided in Table 183. Thirty-four of these samples were also analyzed for hydrocarbon type by Monsanto Method 21-PQ-38-63, and comparative data from both modified ASTM D 2789 and the Monsanto method in weight percents are presented in Table 184. Six other samples were analyzed for Aydrona bon types by ASTM D 2425 in addition to modified ASTM D 2789 (four of the samples were also analyzed by the Monsanto method), and comparative data from all three methods are presented in Table 185

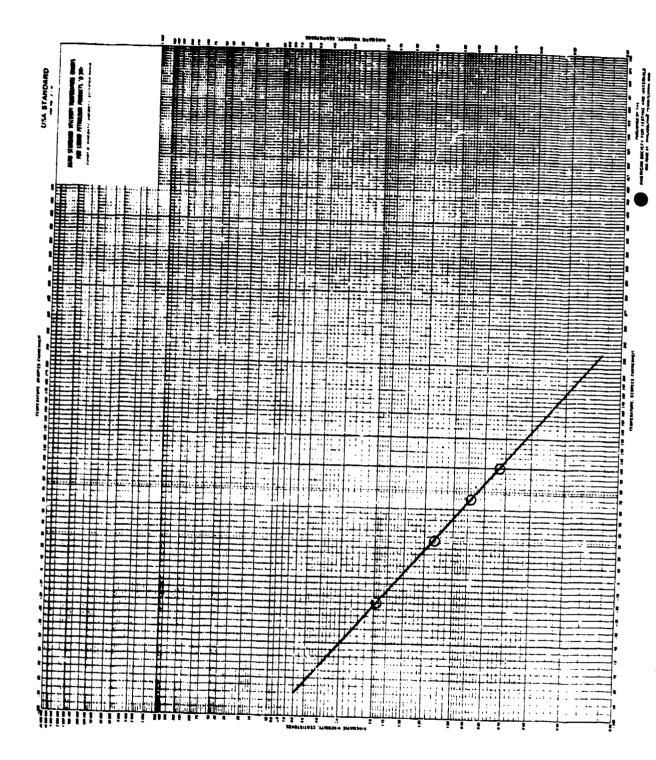
TABLE 176. DENSITY AS A FUNCTION OF TEMPERATURE FOR FUELS TESTED AFTER 1 MAY 1980

	Grams per cubic centimeter			
Sample description	-20°F	32°F	70°F	100°F
1B-792009, JP-4	0.7875	0.7643	0.7475	0.7346
2B-792009, JP-8	0.8440	0.8227	0.8075	0.7955
13B-792009, DF-2	gel	0.8573	0.8427	0.8313
14B-792009, DF-2	gel	0.8871	0.8722	0.8600
Aromatic Blend	_			
8B-792009	0.8618	0.8385	0.8220	0.8086
9B-792009	0.8300	0.8065	0.7900	0.7771
15B-792009	0.8152	0.7925	0.7765	0.7637
GEC-49B-1	0.7891	0.7655	0.7492	0.7355
1C-792009	0.8014	0.7785	0.7619	0.7486
8C-792009	0.8714	0.8492	0.8330	0.8202
9C-792009	0.8393	0.8170	0.8002	0.7875
13C-792009		0.6613	0.8462	0.8340
14C-792009		0.8929	0.8777	0.8655
15C-792009	0.8196	0.7980	0.7824	0.7700
JP-5, Tank 13	0.8503	0.8290	0.8134	0.8016
P&W 79-C-2086, Petrolcum JP-4	0.7938	0.7708	0.7540	0.7411
P&W 79-C-2086, Shale JP-4	0.8146	0.7925	0.7755	0.7637
P&W 79-C-2086, Blend #1	0.8215	0.7997	0.7839	0.7715
P&W 79-C-2086, Blend #2	0.8304	0.8080	0.7915	0.7785
P&W 79-C-2086, Blend #3	0.8336	0.8125	0.7967	0.7843
P&W 79-C-2086, Blend #4	0.8350	0.8099	0.7920	0.7778
P&W 79-C-2009-62B-1, DF-2	0.7911	0.7681	0.7513	0.7376
P&W 792086, M50001	0.8132	0.7918	0.7753	0.7623
P&W 792086, M60001	0.8483	0.8262	0.8097	0.7968
P&W 792086, M70001	0.9119	0.8907	0.8750	0.8624
P&W 792086, M80001	frozen	0.8974	0.8818	0.8695
GEC 792009, 77B	0.7910	0.7681	0.7508	0.7377
GEC 792009, 78B	slushy	0.8699	0.8541	0.8425
GEC 792009, 13C-2	frozen	0.8615	0.8461	0.8340
P&W M50014A-2	0.8500	0.8282	0.8126	0.8008
P&W MJ0016A	0.7952	0.7733	0.7563	0.7434
P&W MJ0013B	0.7970	0.7746	0.7584	0.7447
POSF-D-81-043, GECF-1D, JP-4	0.7910	0.7689	0.7520	0.7390
AEDC, POSF-C-81-134, A-1, JP-4	0.7992	0.7767	0.7598	0.7468
POSF-D-81-044, GECF-13D, DF-2	frozen	0.8588	0.8440	0.8319
POSF-D-81-042, GECF-14D,				
DF-2/ARO	frozen	0.8840	0.8687	0.8571

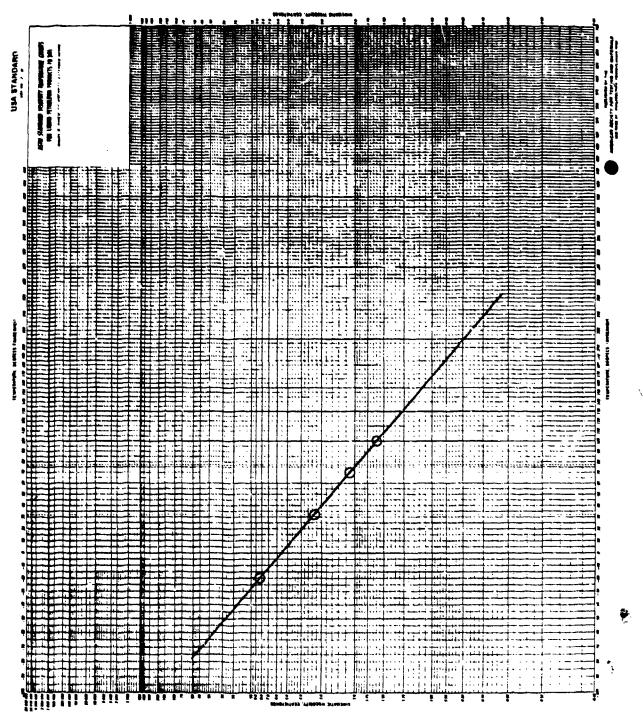
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			ic centime
Sample description	-20°F	32°F	70°F
POSF-D-81-046, GECF-1E, JP-4		0.7797	
POSF-D-81-045, GECF-13E, DF-2 DDP-81-25 Blending JP-4,	frozen	0.8611	0.8458
Tank B-1, 5-5-81 DDP-81-26 DF-2 Base stock,	→ m	0.7684	0.7519
5-5-81	***	0.8639	0.8483
GECS-24-D, Reference JP-4	0.7909		
GECS-26-D, Reference JP-4 DDP-81-08 Blending JP-5,	0.7964	0.7745	0.7577
Tank F-1, 4-23-81	0.8367	0.8241	0.8085
GECS-81B-1, DF-2	frozen	0.8552	
GECS-82B, DF-2	frozen	0.8526	
POSF-81-59, Blend of JP-4,	1102611	0.0540	0.0300
DF-2, 2040 Solvent	0.8556	0.8331	0.8174
AEDC Blending Stocks			
DDP-81-22, Tank B-11, JP-4		0.7684	
DDP-81-23, Tank B-12, JP-4			0.7517
DDP-81-24, Tank B-13, JP-4		0.7683	
DDF-82-20, Tank F-6, JP-5	~-		0.8111
DDP-82-21, Tank F-7, JP-5			0.8120
DDP-81-17, Tank F-9, DF-2			0.8375
DDP-81-18, Tank F-10, DF-2			0.8372
DDP-81,19, Tank F-11, DF-2			0.8374
DDP-81-12, Tank F-8, Xy-B DDP-81-14, Tank B-18, A-400		0.8856	0.8680 0.9611
DDP-81-28, Tank F-12, AEDC		• • • • • • • • • • • • • • • • • • • •	••••
Blend 2 (6 July 81)		0.7950	0.7783
DDP-81-29, Tank F-11, AEDC Blend 3 (6 July 81)		0.8248	0.8093
DDP-81-30, Tank F-7, AEDC			
Blend 5 (6 July 81) DDP-81-31, Tank F-9, AEDC		0.8497	0.8336
Blend 6 (6 July 81)		0.8485	0.8327
E314A, JP-8 Blend #3 (P&W)	0.8318	0.8109	
M50003B, JP-4 Blend #5 (P&W)	0.8501	0.8291	
M50008B, JP-4 Blend 5 (P&W)	0.8489	0.8280	
MS0007A, Shale JP-4 (P&W)	0.8184	0.7970	0.7810
MS0010A, Shale JP-4 (P&W)	0.8169	0.7950	0.7792
MS0011A, Shale JP-4 (P&W)	0.8201	0.7982	0.7820
EJ0020B, JP-4 (P&W)	0.7966	0.7748	0.7574
	396		
	396		

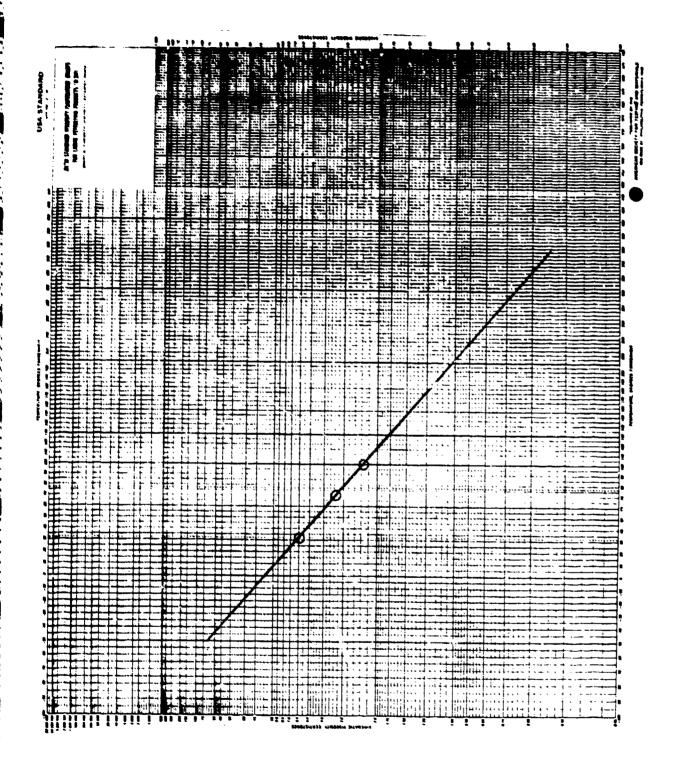


Viscosity/temperature plot for fuel 1B-792009 JP-4. Figure 166.

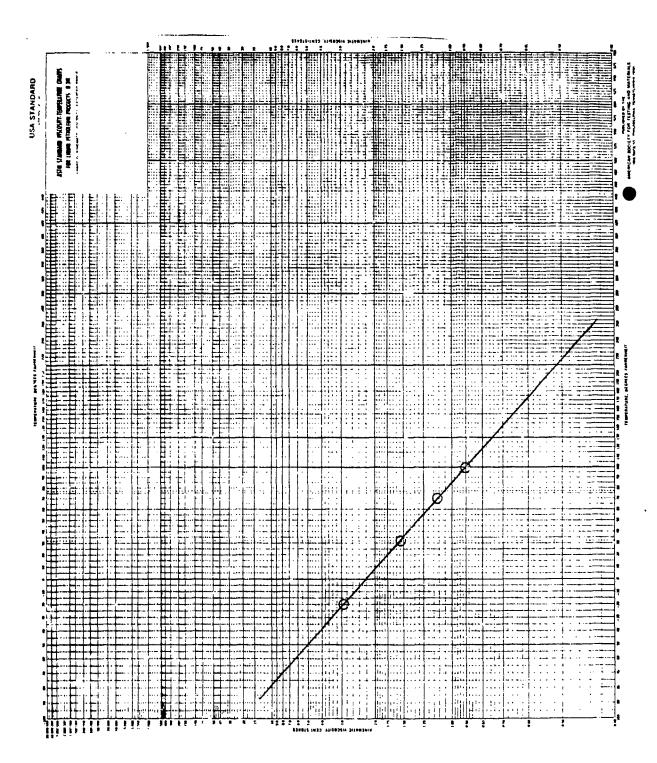


Viscosity/temperature plot for fuel 2B-792009 JP-8. Figure 167.

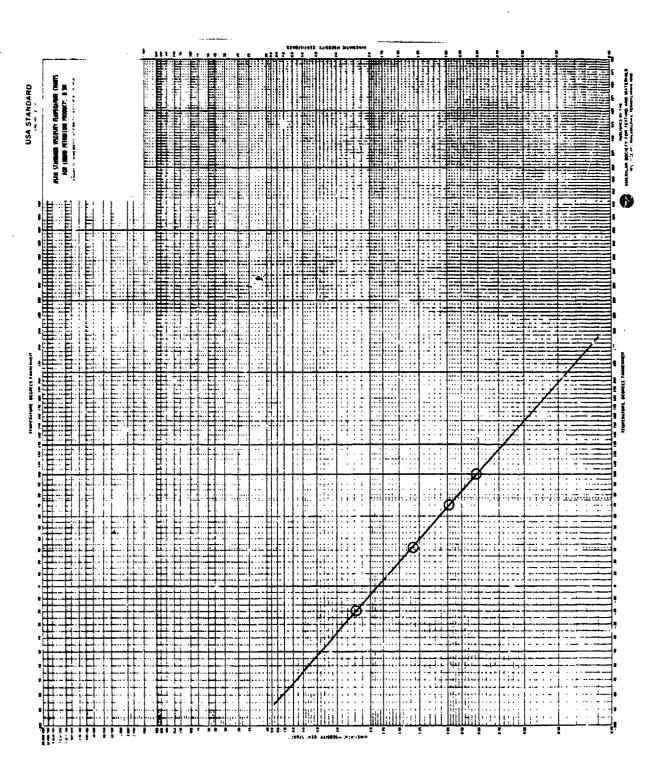
Viscosity/temperature plot for fuel 13B-792009 DF-2 Figure 168.



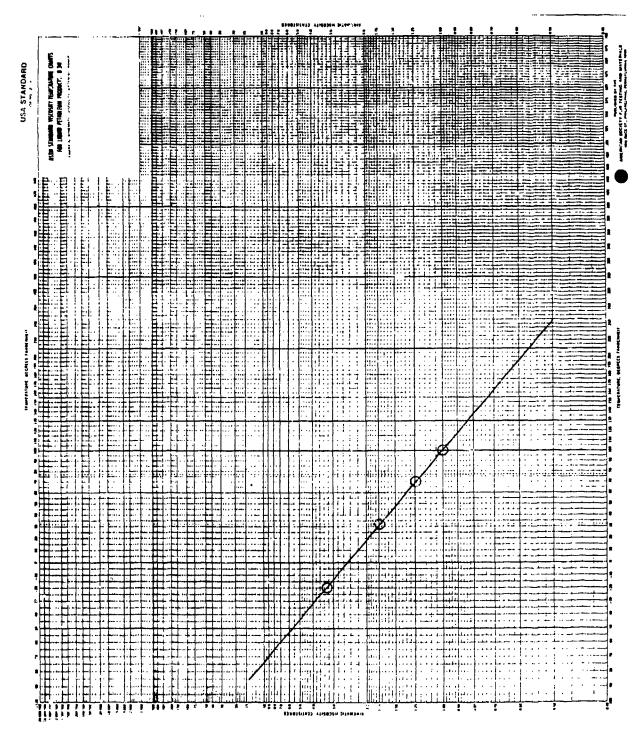
Viscosity/temperature plot for fuel 14B-792009 DF-2 aromatic blend Figure 169.



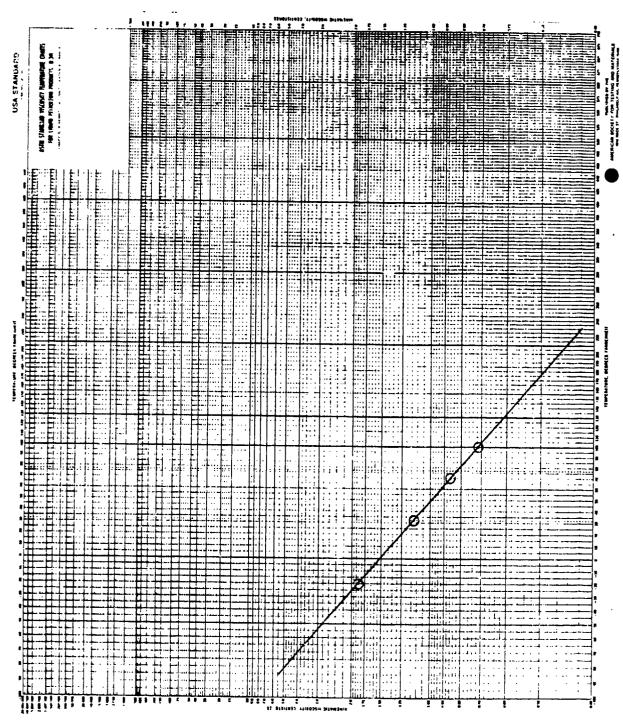
Viscosity/temperature plot for inel 8B-792009



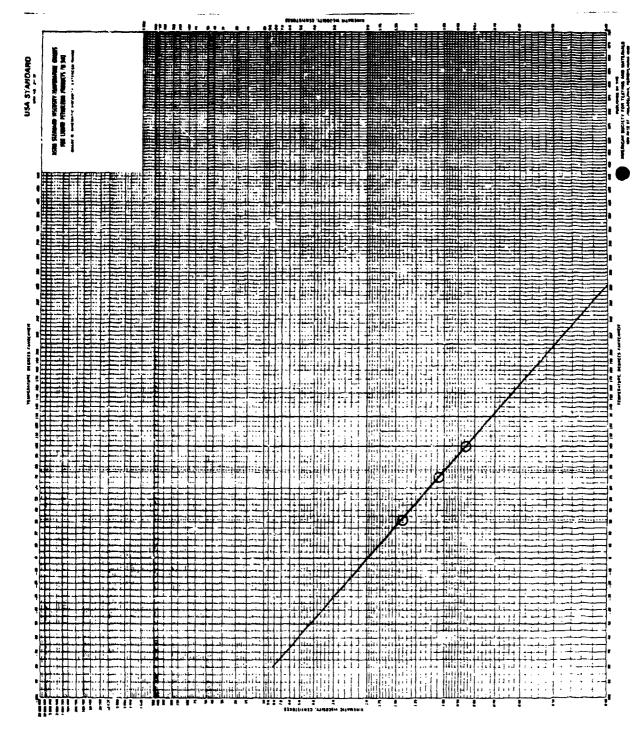
Viscosity/temperature plot for fuel 9B-792009



Viscosity/temperature plot for fuel 15B-792009. Figure 172.

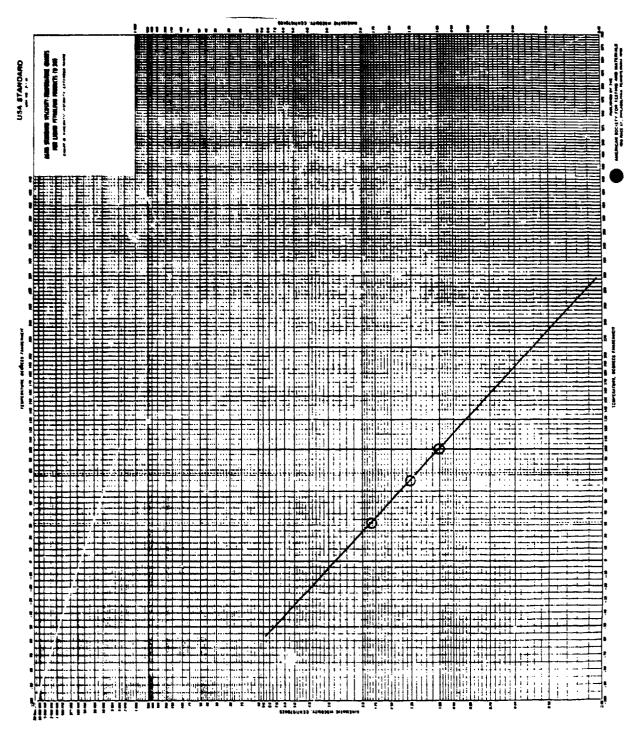


Viscosity/temperature plot for fuel GEC-49B-1 Figure 173.

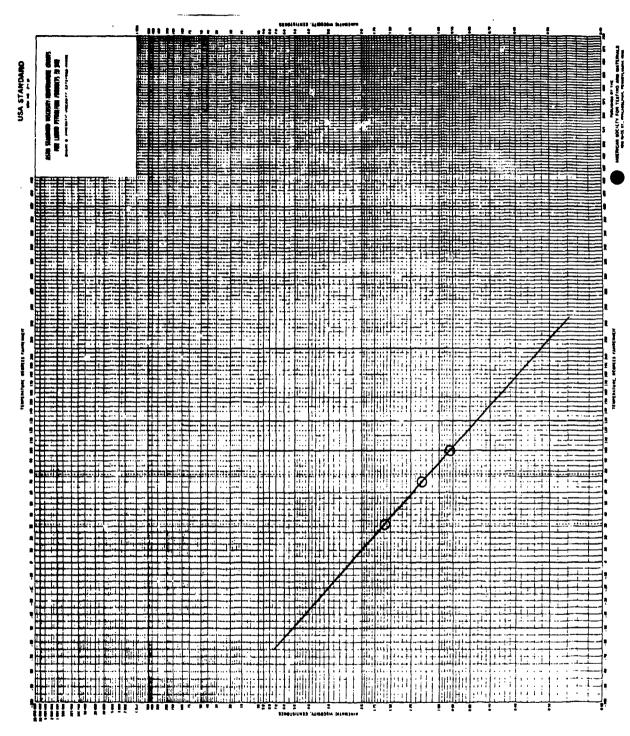


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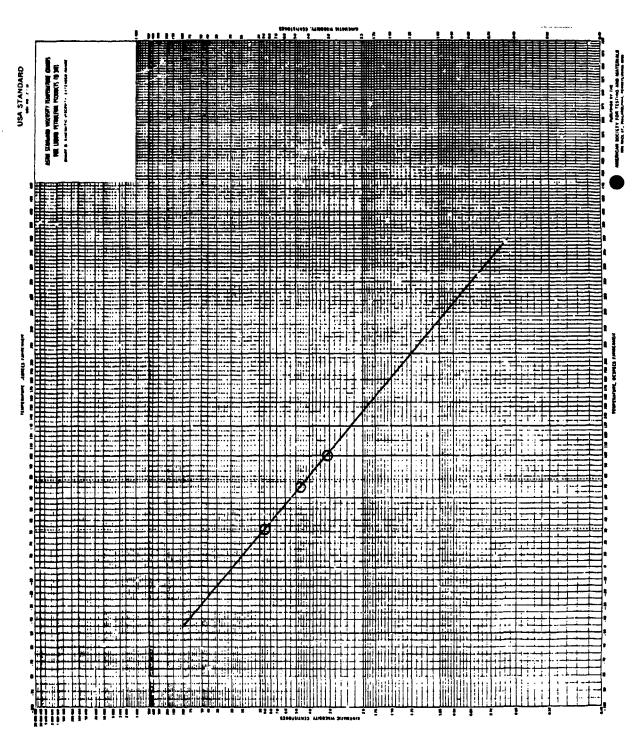
Viscosity/temperature plot for fuel 1C-792009.



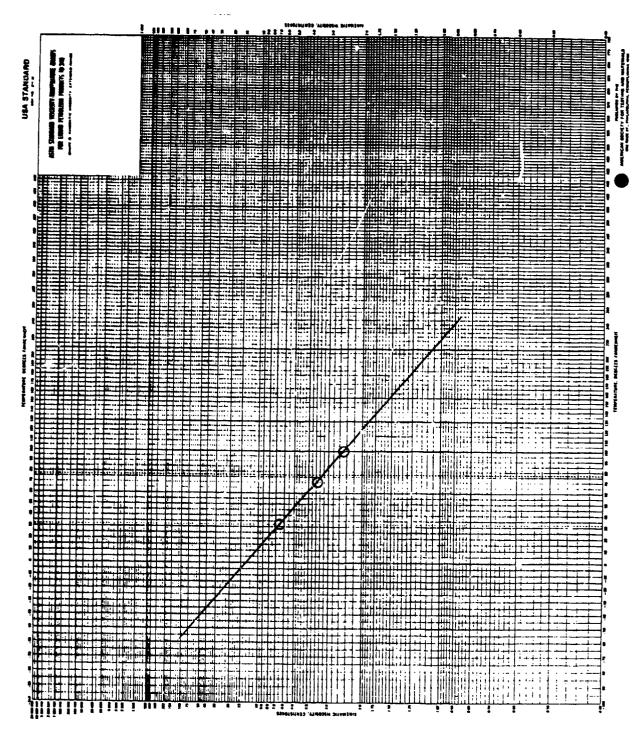
Viscosity/temperature plot for fuel 8C-792009.



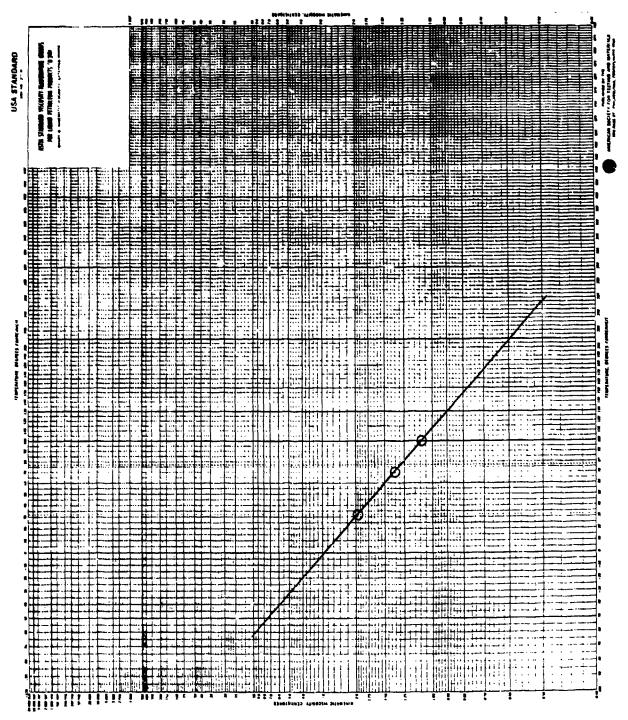
Viscosity/temperature plot for fuel 9C-792009.



Viscosity/temperature plot for fuel 13C-792009

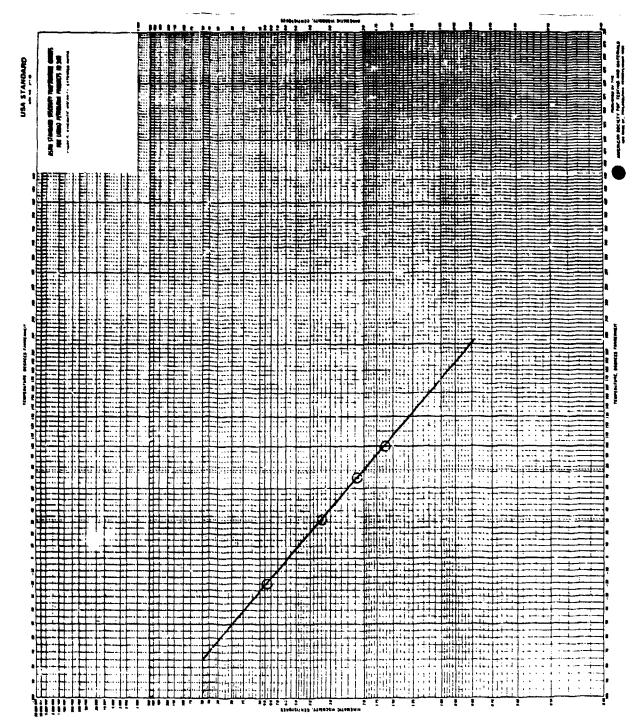


Viscosity/temperature plot for fuel 14C-792009 Figure 178.



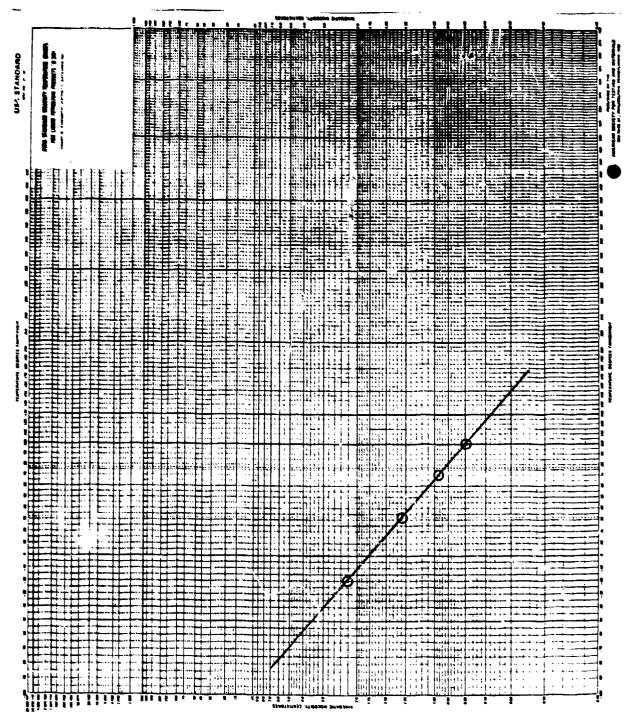
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Viscosity/temperature plot for fuel 15C-792009

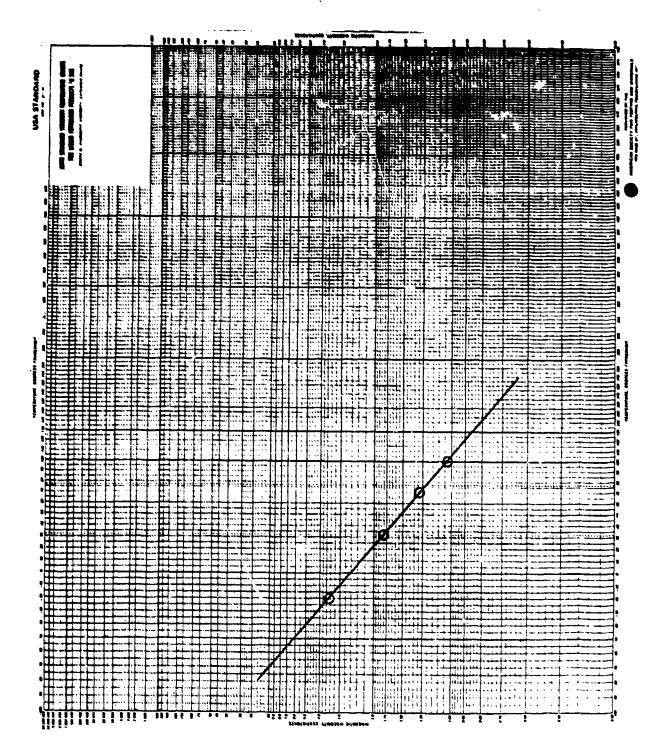


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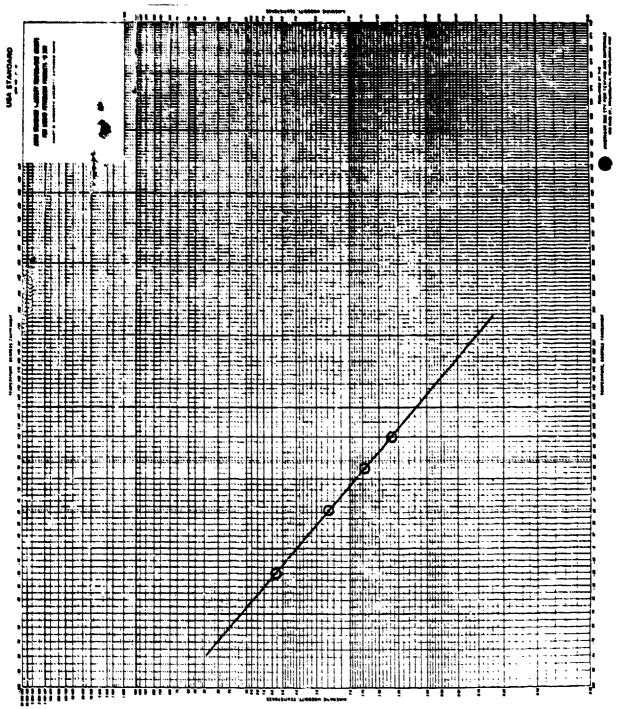
Viscosity/temperature plot for fuel JP-5, Tank 13 Figure 180.



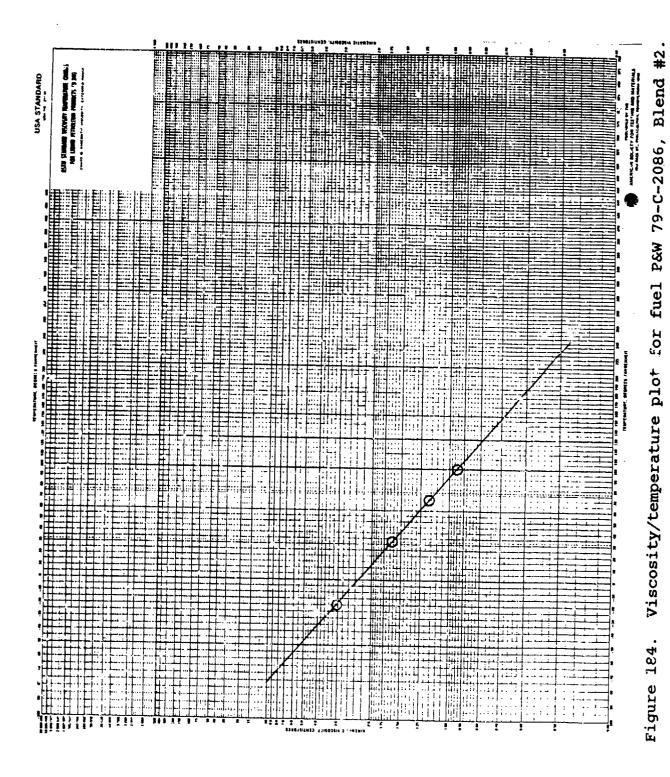
Viscosity/temperature plot for fuel P&W 79-C-2086, Petroleum JP-4 Figure 181.

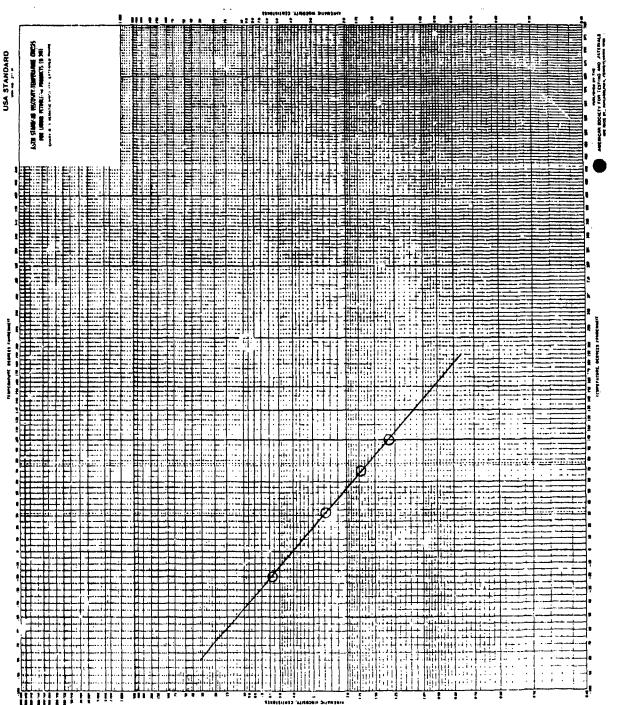


Viscosity/temperature plot for fuel P&W 79-C-2086, Shale JP-4. Figure 132.

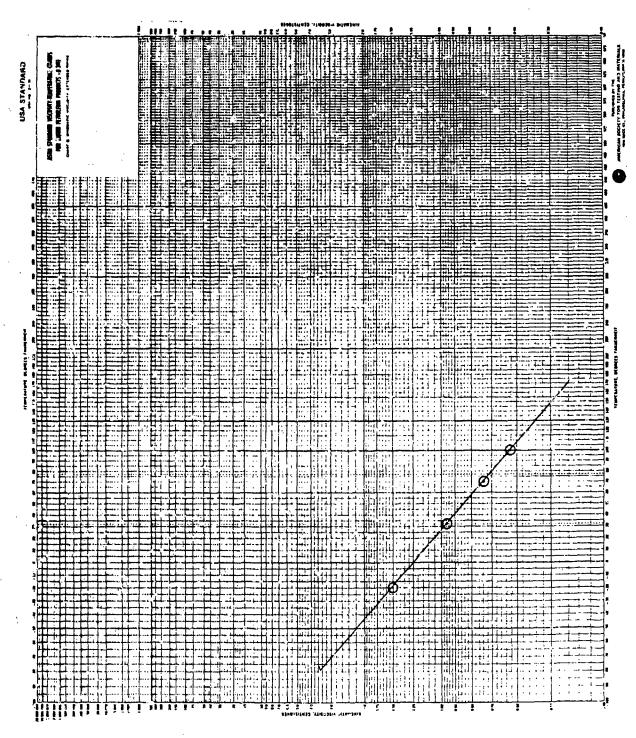


Viscosity/temperature plot for fuel P&W 79-C-2086, Blend #1 Figure 183.

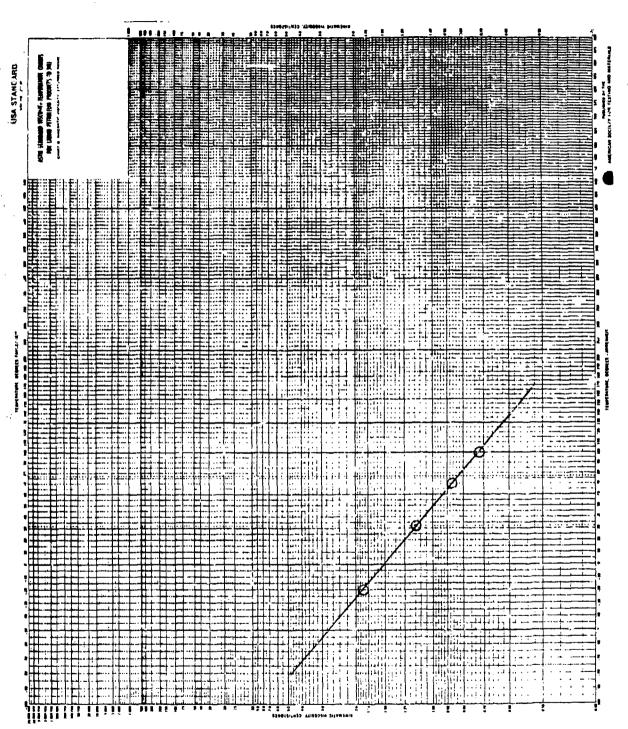




Viscosity/temperature plot for fuel P&W 79-C-2086, Blend #3. Figure 185.



Viscosity/temperature plot for fuel P&W 79-C-2086, Blend #4 Figure 186.



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Viscosity/temperature plot for fuel GECS-62B-1 Figure 187.

Figure 188. Viscosity/temperature plot for fuel M50001.

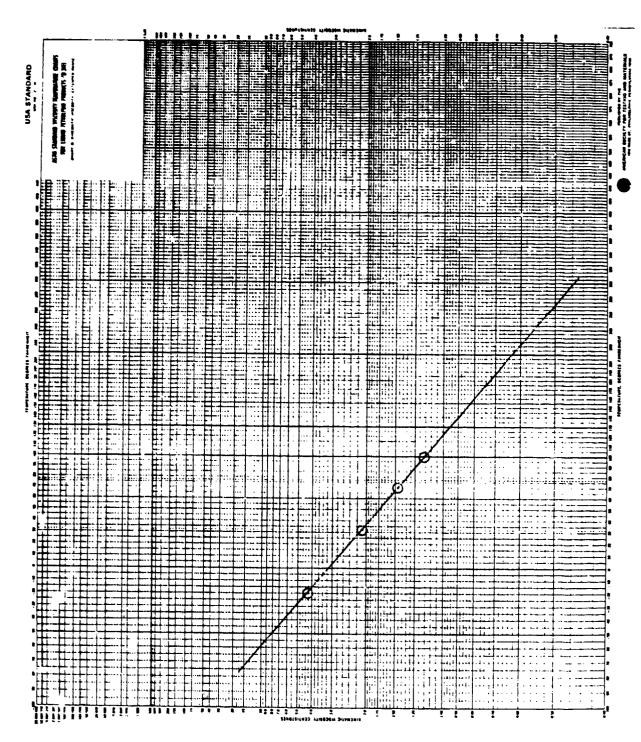
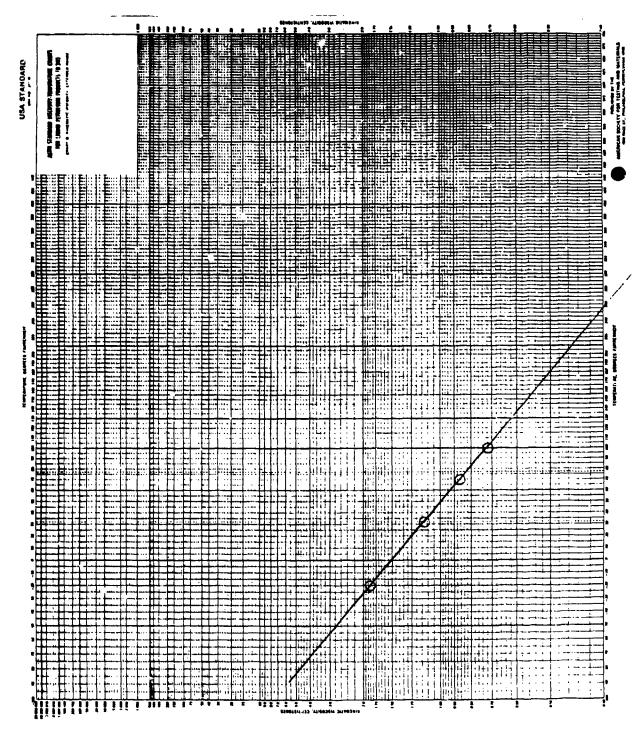


Figure 189. Viscosity/temperature plot for fuel M60001.



igure 190. Viscosity/temperature plot for fuel M70001.

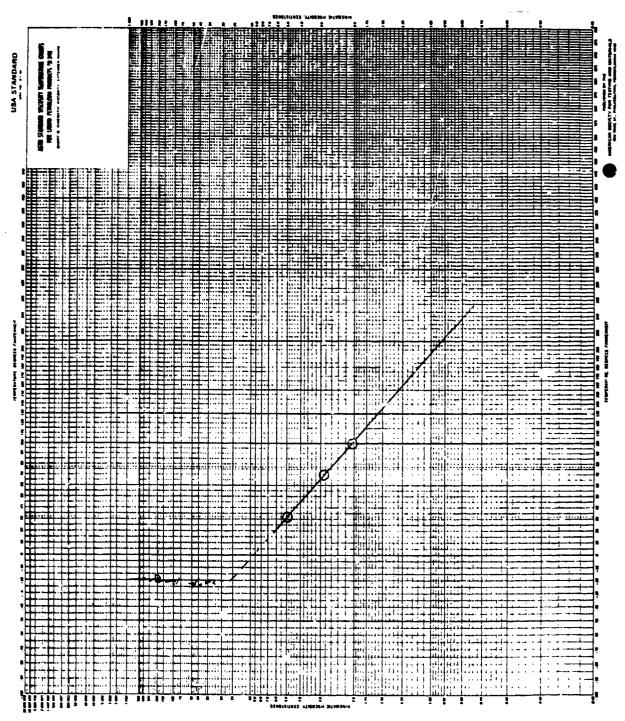
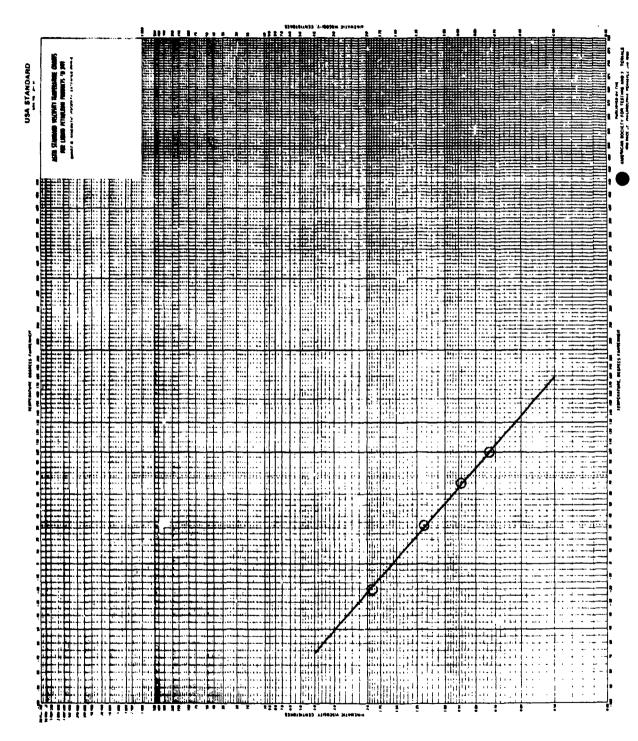
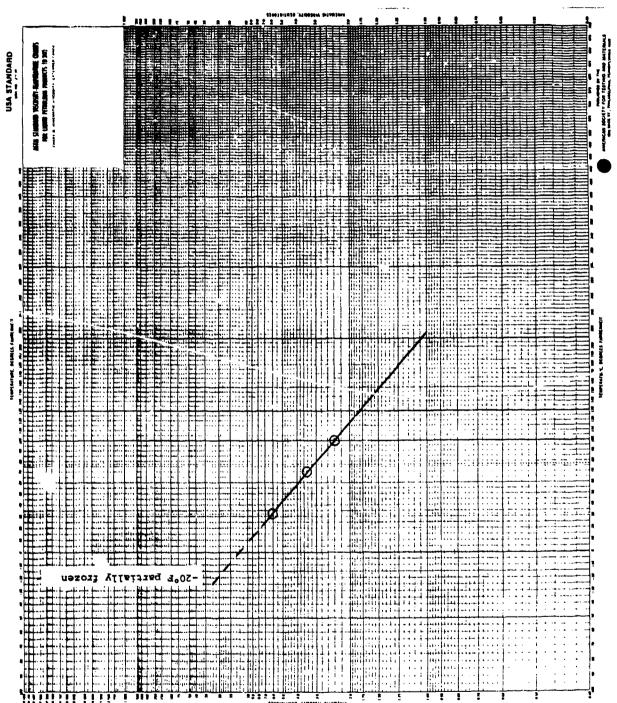


Figure 191. Viscosity/temperature plot for fuel M80001.



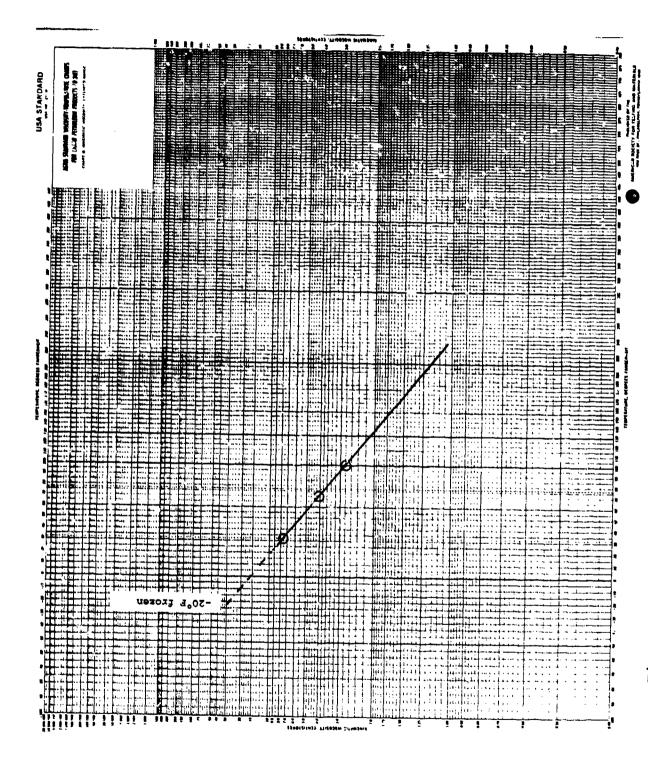
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Viscosity/temperature plot for fuel GEC-77B-792009 Figure 192.

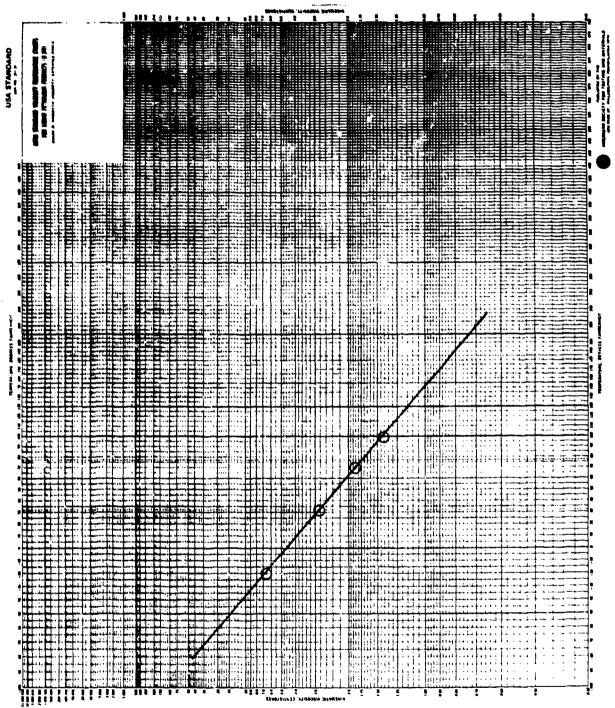


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Viscosity/temperature plot for fuel GEC-78B-792009. Figure 193.



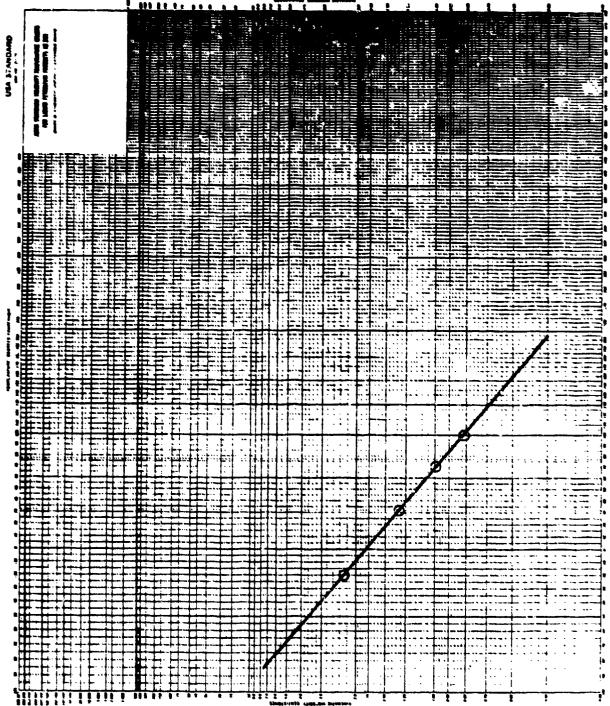
Viscosity/temperature plot for fuel 13C-2-792009. Figure 194.



Viscosity/temperature plot for fuel M50014A-2 Figure 195.

Viscosity/temperature plot for fuel MJ0016A

Figure 196.



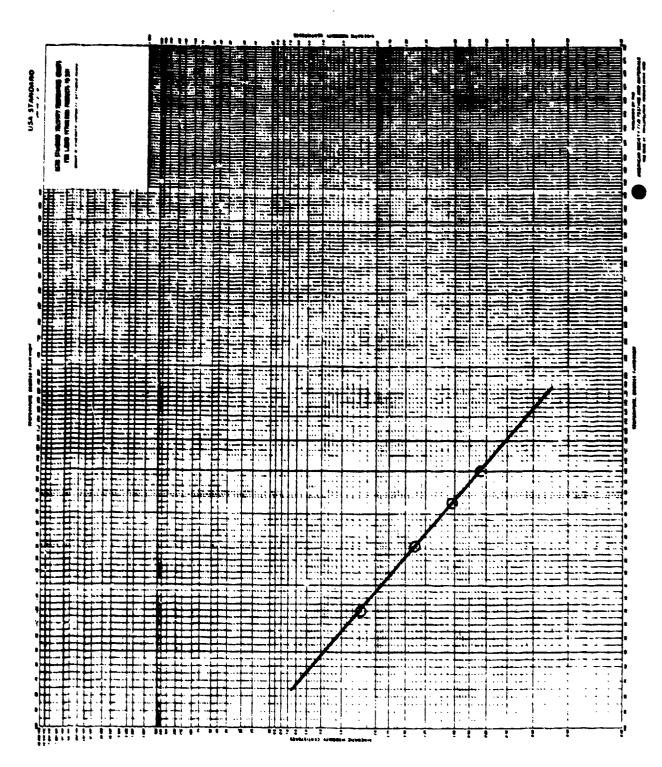
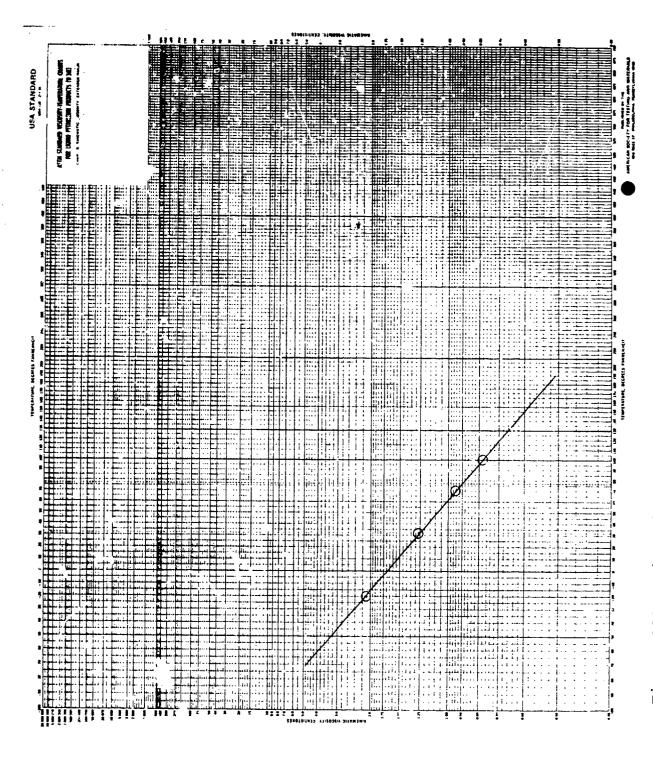
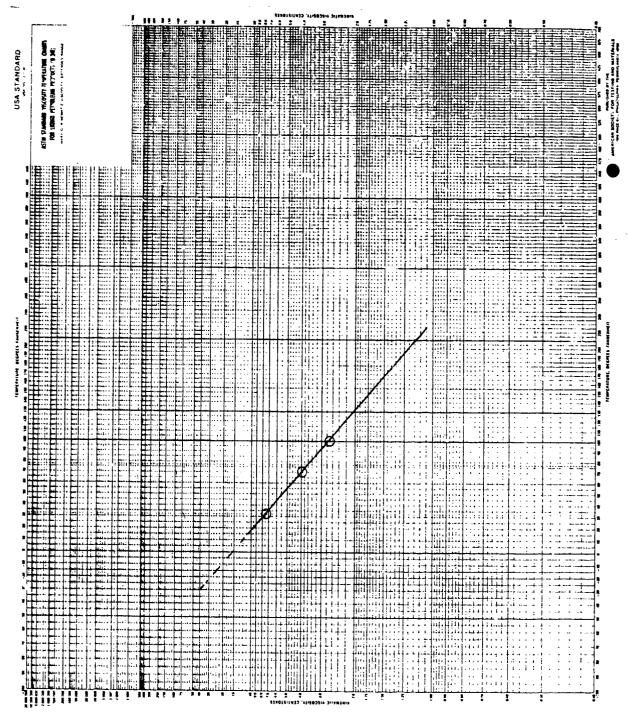


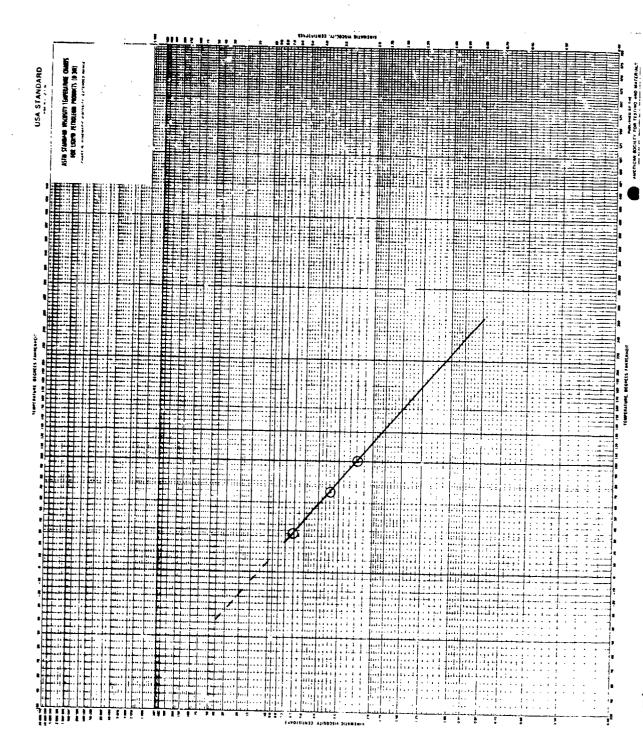
Figure 197. Viscosity/temperature plot for fuel MJ0013B.



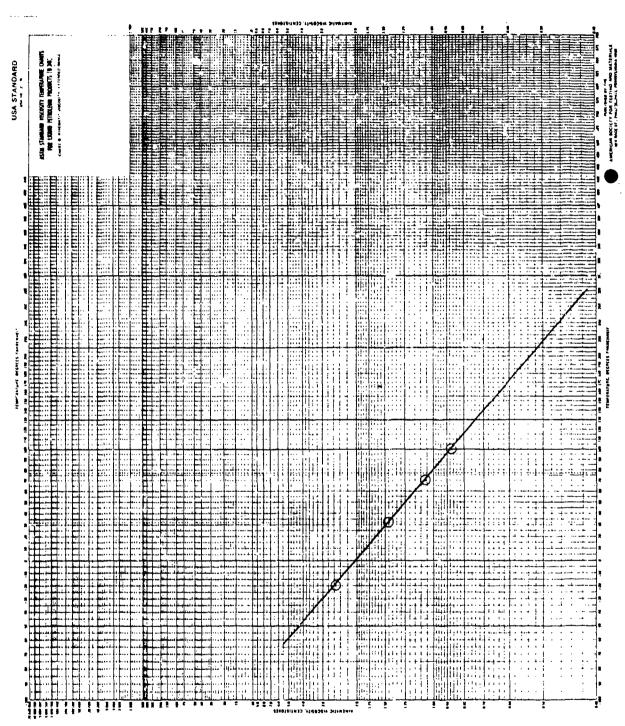
Viscosity/temperature plot for fuel GECF-1D, (JP-4)-043



Viscosity/temperature plot for fuel GECF-13D, (DF-2)-044 Figure 199.

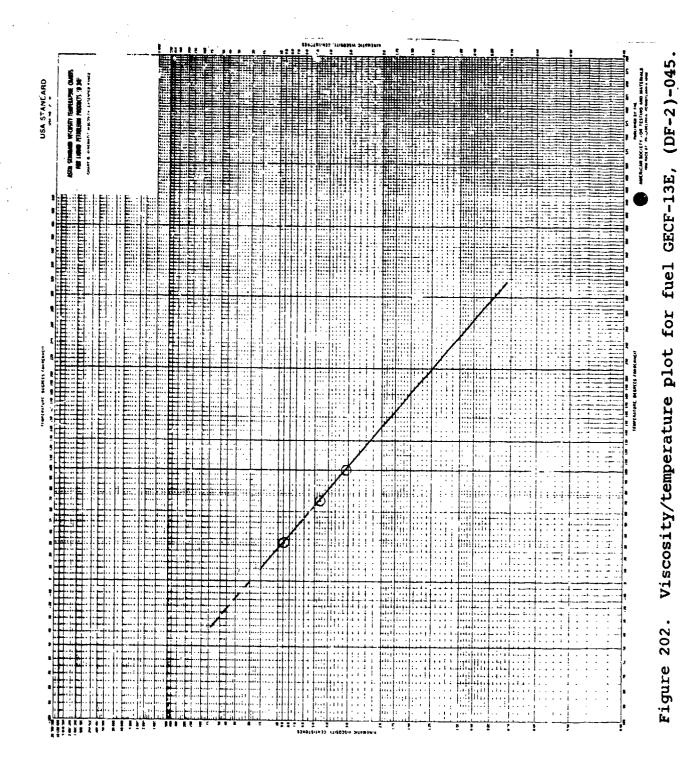


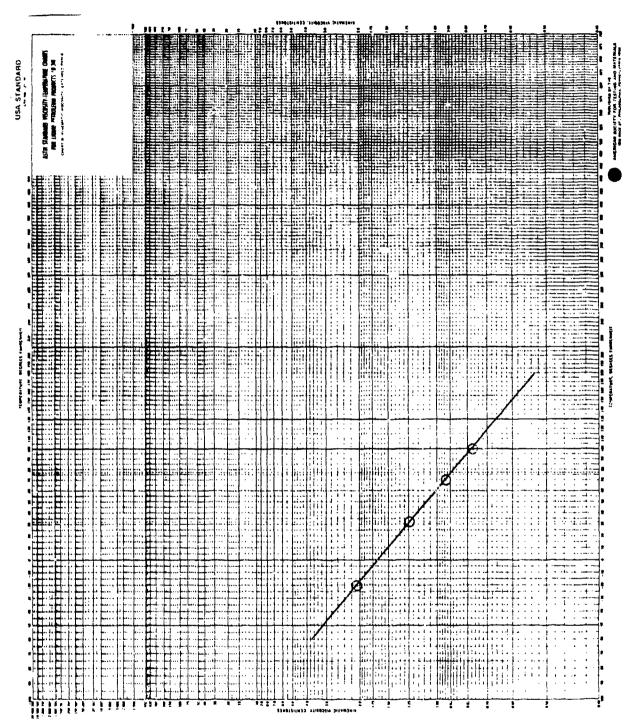
Viscosity/temperature plot for fuel GECF-14D POSF-D-81-042



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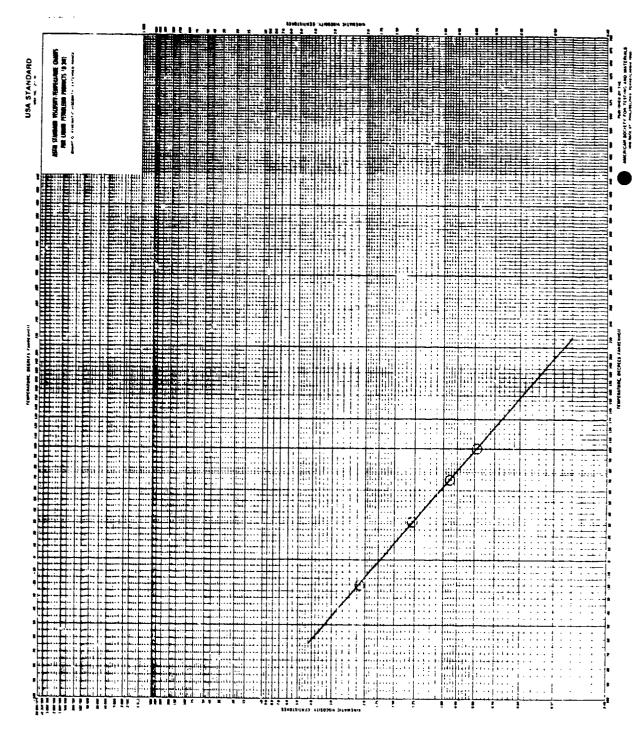
Viscosity, temperature plot for fuel GECF-1E, (JP-4)-046. Figure 201.



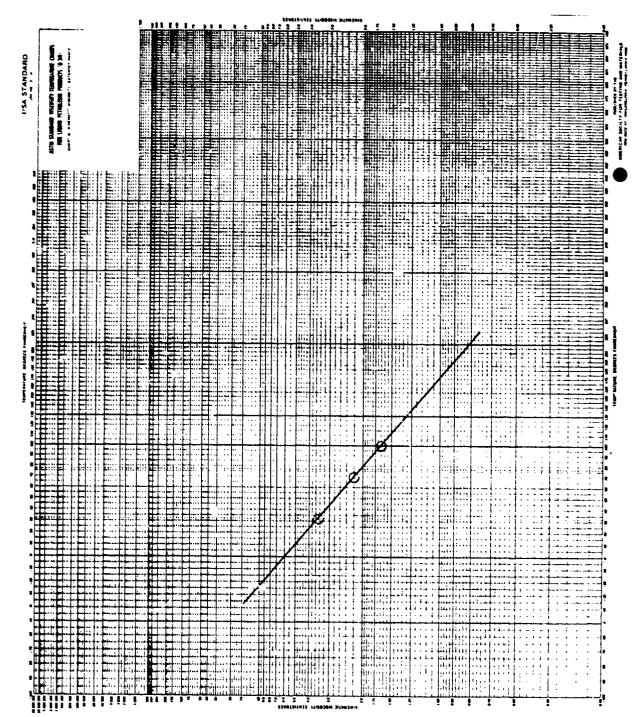


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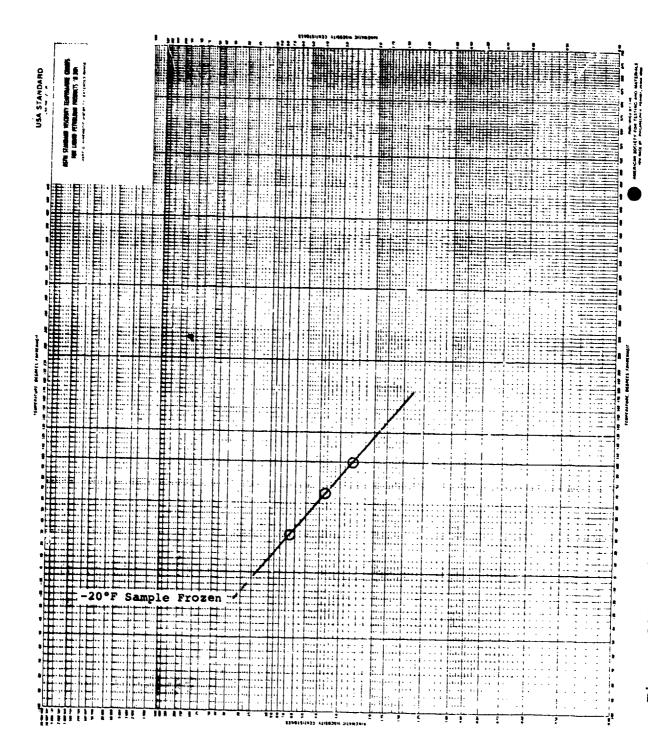
Viscosity/temperature plot for fuel GECS-24D Figure 203.



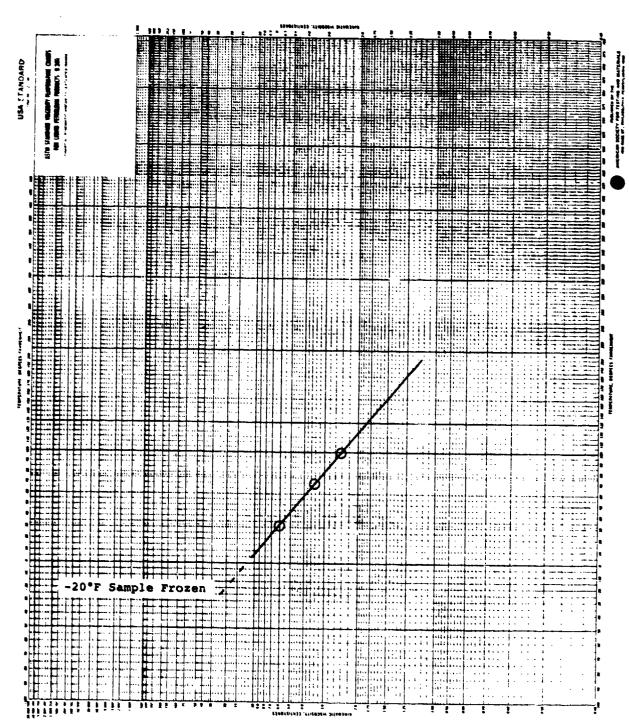
gure 204. Viscosity/temperature plot for fuel GECS-26D.



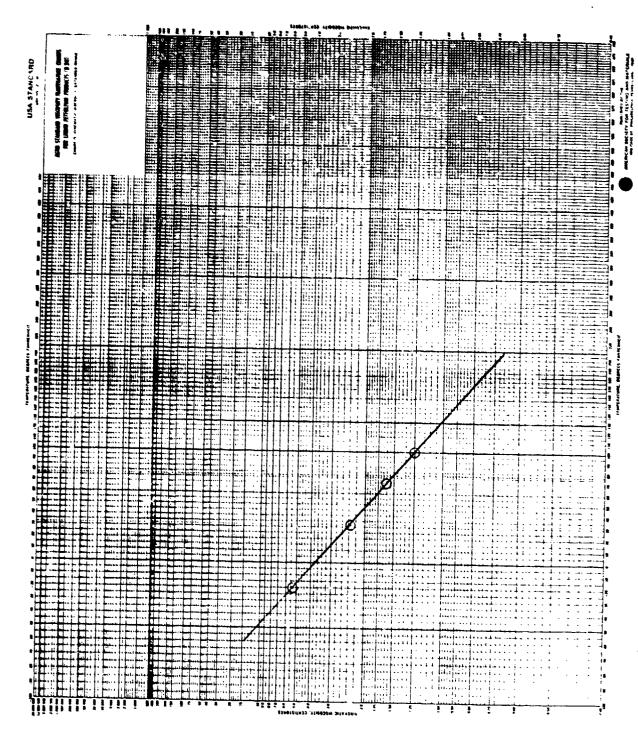
Viscosity/temperature plot for fuel DDP-81-08



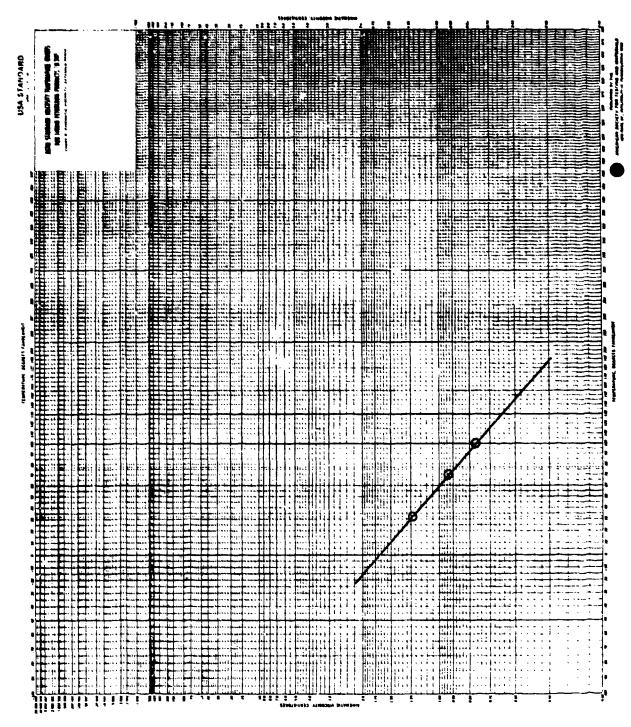
Viscosity/temperature plot for fuel GECS-81B-1 (DF-2)



Viscosity/temperature plot for fuel GECS-82B (DF-2) Figure 207.

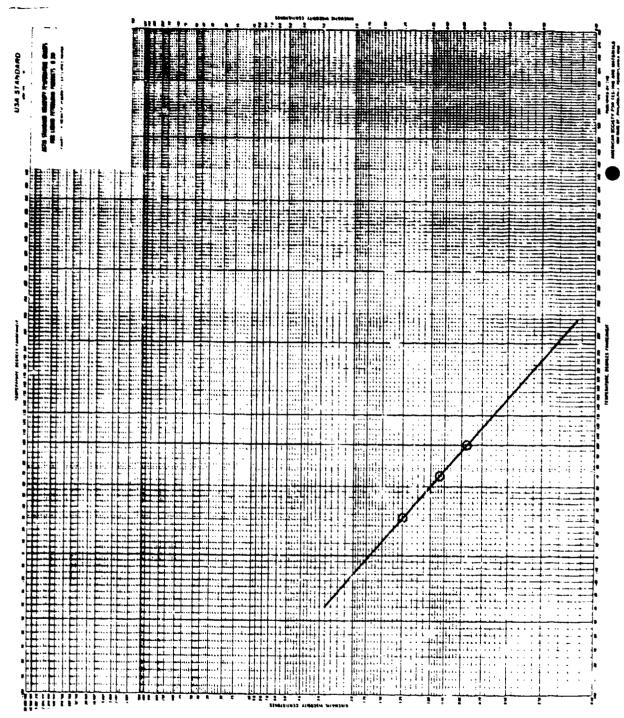


Viscosity/temperature plot for fuel POSF-D-81-59

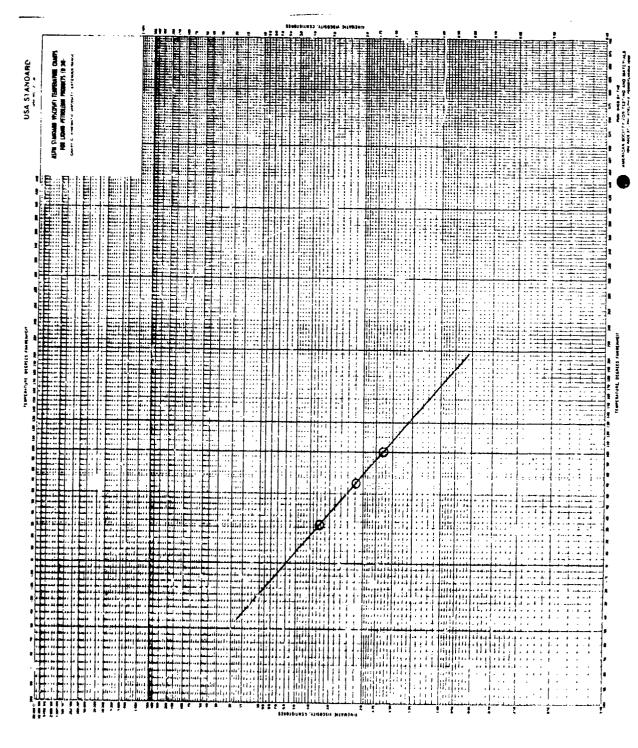


Viscosity/temperature plot for fuel JP-4, Tank B-11: DDP-81-22 Figure 209.

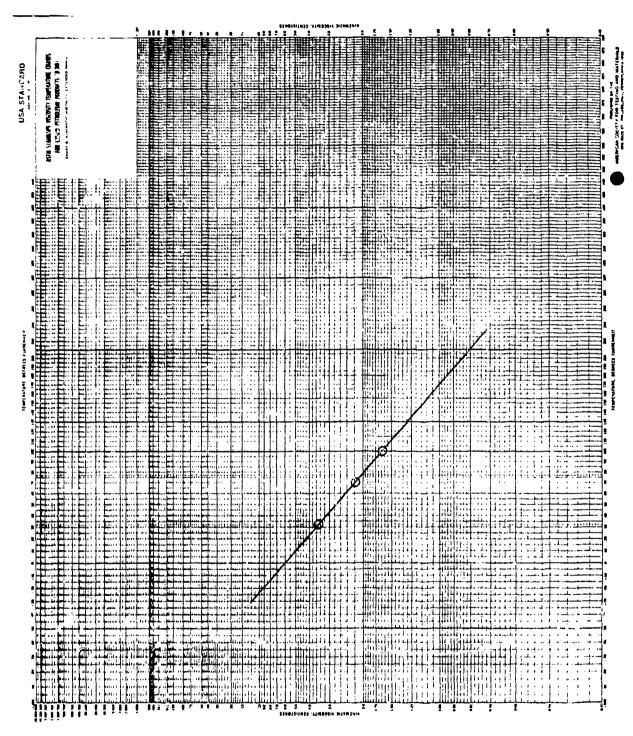
Viscosity/temperature plot for fuel JP-4, Tank B-12: DDP-81-23 Figure 210.



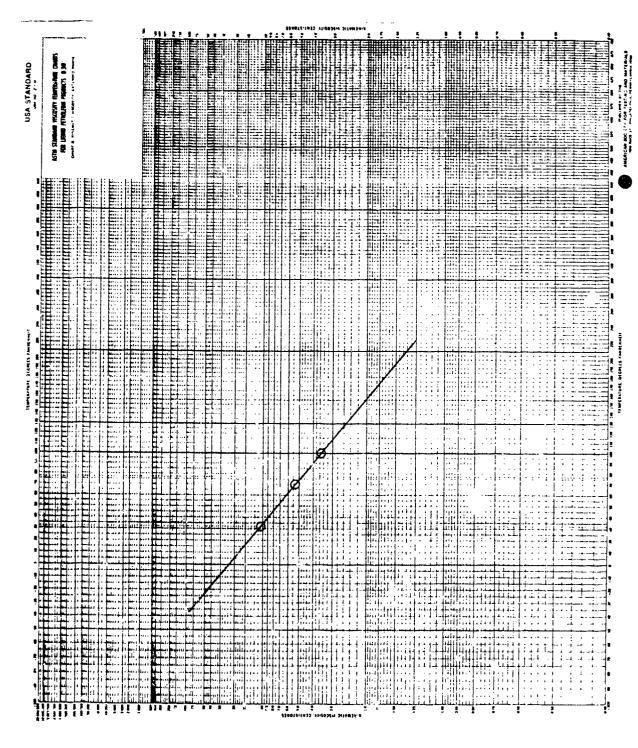
DDP-81-24 Viscosity/temperature plot for fuel J2-4, Tank B-13: Figure 211.



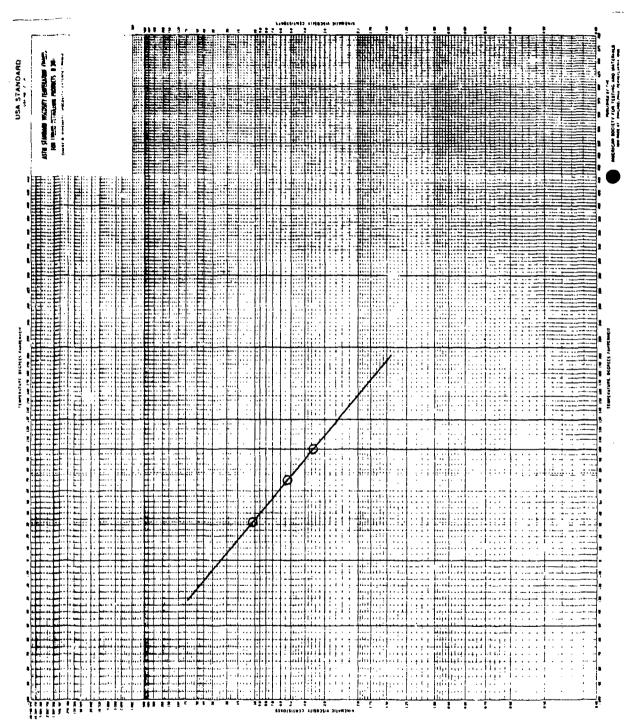
DDF-81-20 Viscosity/temperature plot for fuel JP-5, Tank F-6: Figure 212.



DDP-81-21 Tank F-7: Viscosity/temperature plot for fuel JP-5, Figure 213.

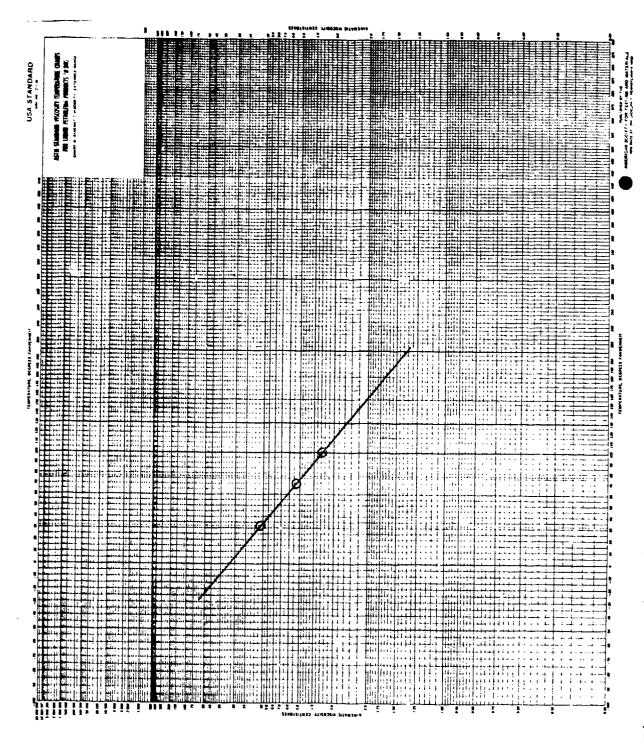


Viscosity/temperature plot for fuel DF-2, Tank F-9: Figure 214.



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DDP-81-18 Viscosity/temperature plot for fuel DF-2, Tank F-10: Figure 215.



DDP-81-19 Viscosity/temperature plot for fuel DF-2, Tank F-11: Figure 216.

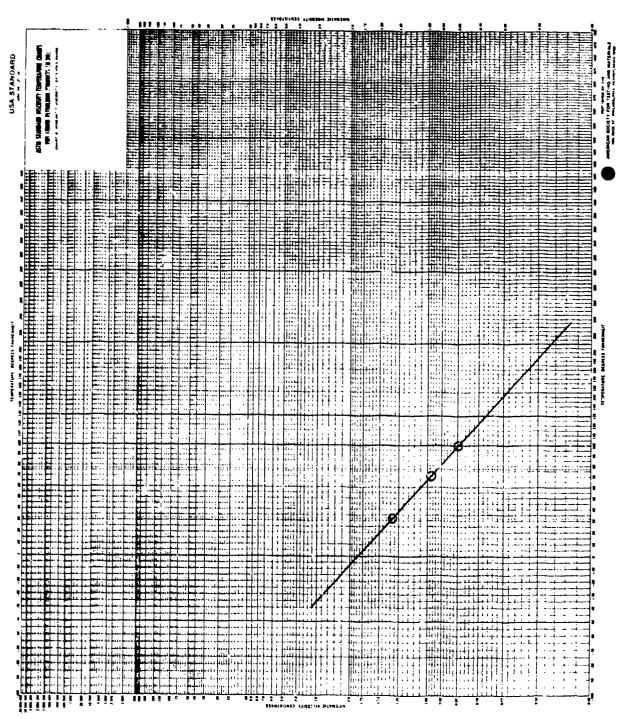


Figure 217. Viscosity/temperature plot for fuel XY-B, Tank F-8: DDP-81-12, Xylene bottoms.

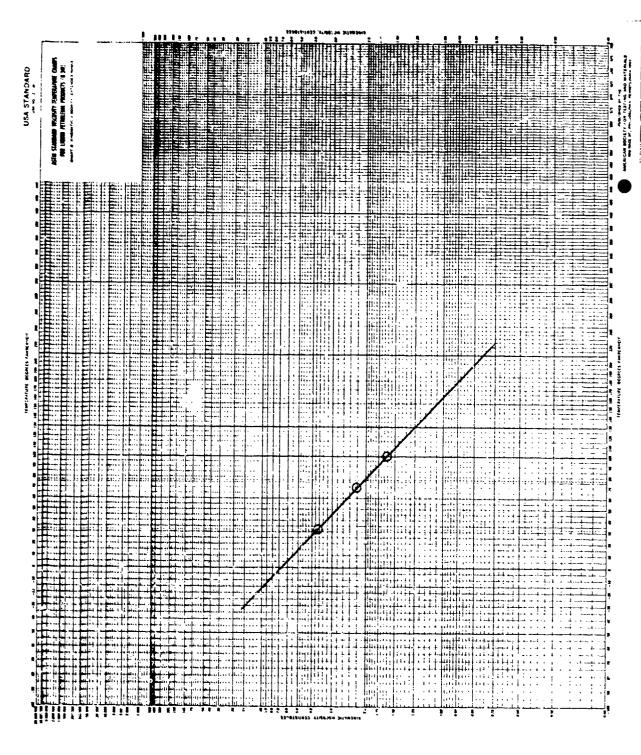
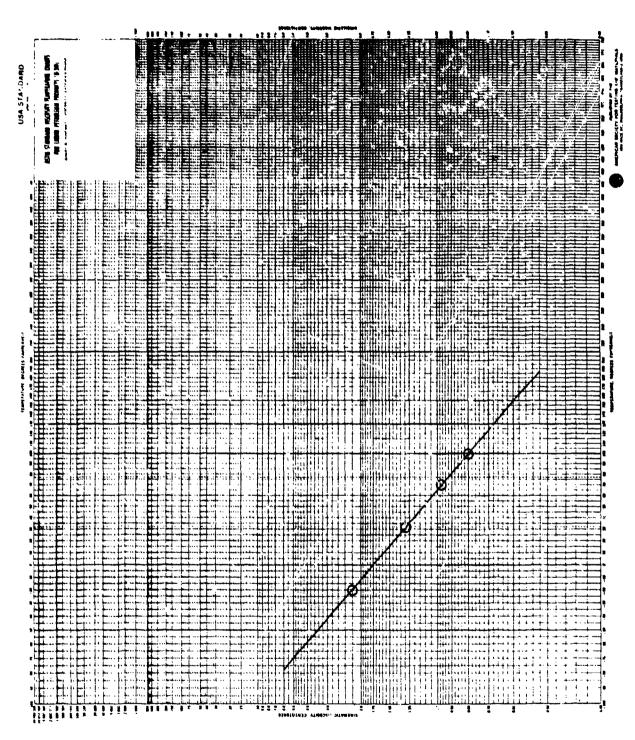


Figure 218. Viscosity/temperature plot for fuel A-400, Tank B-18: DDP-81-14, (Getty A-400).



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Figure 219. Viscosity/temperature plot for fuel POSF-C-81-134, AEDC JP-4, 1-A.

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TABLE 177. KINEMATIC VISCOSITY VS. TEMPERATURE FOR FUELS TESTED AFTER 1 MAY 80

·		5	-41	
	000		stokes	
Sample description	-20°F	32°F	70°F	100°F
18 502000 TB 4	1 041	1 150	0.751	0 7060
1B-792009, JP-4	1.941			0.7262
2B-792009, JP-8	8.360		2.129	
13B-792009, DF-2	gel	7.541	4.083	2.794
14B-792009, DF-2	_			
Aromatic blend	gel			
8B-792009	2.921	1.539		0.9005
9B-792009	2.345		0.9856	
15B-792009	3.242		1.241	
GEC-49B-1	1.857			0.7172
1C-792009	2.55	1.405	1.035	0.8453
8C-792009	3.80	1.807	1.258	1.004
9C-792009	3.00	1.582	1.139	0.9225
130-792009		8.945	4.614	3.109
14C-792009		6.537	3.568	2.472
15C-792009	4.00		1.362	1.085
JP-5, Tank 13	8.535	3.362	2.140	1.605
P&W 79-C-2086, Petroleum JP-4	2.220			0.7956
P&W 79-C-2086, Shale JP-4	3.461	-		
P&W 79-C-2086, Blend #1	5.564			
P&W 79-C-2086, Blend #2	3.029			0.9415
P&W 79-C-2086, Blend #3	5.684			
P&W 79-C-2086, Blend #4	1.503			0.5216
GEC 79-C-2009-62B-1, DF-2	1.848			0.7212
P&W 792086, M50001	3.333			
P&W 792086, M60001	4.246	2.086		
P&W 792086, M70001	12.61		2.646	
P&W 792086, M80001	frozen			
GEC 792009, 77B	1.910	1.176		0.7312
· ·	~33			
GEC 792009, 78B				
GEC 792009, 13C-2	frozen			
P&W M5G014A	6.562			
P&W MJ0016A	2.301			0.8148
P&W MJ0013B	2.382	1.371	1.015	
POSF-D-81-043, GECF-1D, JP-4		1.258		0.7766
POSF-D-81-044, GECF-13D, DF-2	frozen	7.266	3.949	2.723
POSF-D-81-042, GECF-14D,				
DF-2/ARO	frozen		3.382	2.366
POSF-D-81-046, GECF-1E, JP-4	2.554			
POSF-D-81-045, GECF-13E, DF-2	frozen			
GECS-24D, Reference JP-4	2.076	1.246		0.7714
GECS-26D, Referer > JP-4	2.132	1.277	0.9520	0.7855
DDP-81-08 Blending JP-5,				
Tank F-1, 4-23-81	5.756	3.479	2.207	1.651

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TABLE 177 (continued)

		Centi	stokes	
Sample description	-20°F	32°F	70°F	100°F
GECS-81B-1, DF-2	frozen	6.642	3.681	2.556
GECS-82B, DF-2	frozen	6.064	3.444	2.416
POSF-D-81-59, Blend of JP-4,				
DF-2, 2040 Solvent	5.438	2.355	1.604	1.252
AEDC Blending Stocks				
DDP-81-22, Tank B-11, JP-4		1.238	0.9357	0.7720
DDP-81-23, Tank B-12, JP-4		1.241	0.9339	0.7728
DDP-81-24, Tank B-13, JP-4		1.249	0.9366	0.7732
DDP-81-20, Tank F-6, JP-5		3.459	2.192	1.639
DDP-81-21, Tank F-7, JP-5		3.463	2.192	1.638
DDP-81-17, Tank F-9, DF-2		10.55	5.355	3.541
DDF-81-18, Tank F-10, DF-2		10.60	5.348	3.545
DDP-81-19, Tank F-11, DF-2		10.66	5.355	3.548
DDP-81-12, Tank F-8, Xy-B		1.327	0.9767	0.7978
DDP-81-14, Tank B=18, A-400		3.656	2.217	1.621
AEDC POSF-C-81-134, A-1 JP-4	2.209	1.293	0.9715	0.7997

TABLE 178. SURFACE TENSION VS. TEMPERATURE OF FUELS TESTED AFTER 1 MAY 80

	D	ynes per	centimete	r
Sample designation	-20°Fª	32°F	70°F	100°F
1B-79009, JP-4	27.28	24.31	22.16	20.43
2B-792009, JP-3	31.33	28.64	26.69	25.15
13B-792009, DF-2	gel	30.37	28.42	26.88
14B-792009, DF-2	-			
Aromatic blend	gel	31.35	29.43	27.88
8B-792009	29.05	26.58	24.75	23.33
9B-792009	28.18	25.82	24.06	22.69
15B-792009	29.05	26.43	24.53	23.01
GEC-49B-1	27.53	24.68	22.53	20.89
1C-792009	27.50	24.73	22.80	21.25
8C-792009	29.58	26,99	25.09	23.61
9C-792009	28.82	26.12	24.12	22.56
13C-792009		30.37	28.55	27.10
14C-792009		31.80	29.98	28,54
15C-792009	29.10	26.60	24.78	23.34
JP-5, Tank 13	30.91	28.34	26.45	24.97

(continued)

P& P& P& P& P& P& P& P& P& P& P& P& P& P	Sample designation W 79-C-2086, Petroleum JP-4 W 79-C-2086, Shale JP-4 W 79-C-2086, Blend #1 W 79-C-2086, Blend #2 W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 77B C 792009, 78B C 792009, 78B C 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B DSF-D-81-043, GECF-1D, JP-4 DSF-D-81-044, GECF-13D, DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	27.39 29.27 29.58 29.94 30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	32°F 24.72 26.54 26.78 27.07 27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74 25.14	70°F 22.80 24.53 24.69 24.94 25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84 23.08	100 21 22 23 23 24 22 20 22 23 23 23 27 20 26 26 24 21
P& P& P& P& P& P& P& P& P& P& P& P& P& P	W 79-C-2086, Petroleum JP-4 W 79-C-2086, Shale JP-4 W 79-C-2086, Blend #1 W 79-C-2086, Blend #2 W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W MJ0013B DSF-D-81-043, GECF-1D, JP-4 DSF-D-81-044, GECF-13D, DF-2 DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	27.39 29.27 29.58 29.94 30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	24.72 26.54 26.78 27.07 27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	22.80 24.53 24.69 24.94 25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	21 22 23 23 24 22 20 22 23 23 27 20 26 24 21
P& P& P& P& P& P& P& P& P& P& P& P& P& P	W 79-C-2086, Shale JP-4 W 79-C-2086, Blend #1 W 79-C-2086, Blend #2 W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 77B C 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B DSF-D-81-043, GECF-1D, JP-4 DSFD-81-044, GECF-13D, DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	29.27 29.58 29.94 30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	26.54 26.78 27.07 27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	24.53 24.69 24.94 25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	22 23 24 22 20 22 23 23 27 20 26 26 24 21
P& P& P& P& P& P& P& P& P& P& P& P& P& P	W 79-C-2086, Blend #1 W 79-C-2086, Blend #2 W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B DSF-D-81-043, GECF-1D, JP-4 DSF-D-81-044, GECF-13D, DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	29.58 29.94 30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	26.78 27.07 27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	24.69 24.94 25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	23 24 22 20 22 23 23 27 20 26 26 24 21
P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&	W 79-C-2086, Blend #2 W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSF-D-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	29.94 30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	27.07 27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	24.94 25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	23 24 22 20 22 23 23 27 20 26 24 21
P& P& P& P& P& P& P& P& P& P& P& P& P& P	W 79-C-2086, Blend #3 W 79-C-2086, Blend #4 C 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSF-D-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	30.65 28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	27.93 25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	25.93 23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	24 22 20 22 23 23 27 20 26 24 21
P& P& P& P& P& P& P& P& PO PO PO GE DD	W 79-C-2086, Blend #4 CC 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M80001 CC 792009, 77B CC 792009, 78B CC 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSF-D-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	28.33 28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	25.61 25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	23.62 22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	22 20 22 23 23 27 20 26 26 24 21
P& P& P& P& P& P& P& P& P& P& P& P& P& P	CC 79-C-2009-62B-1, DF-2 W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 CC 792009, 77B CC 792009, 78B CC 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B DSF-D-81-043, GECF-1D, JP-4 DSFD-81-044, GECF-13D, DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	28.82 29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	25.41 26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	22.93 24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	20 23 23 27 20 26 26 24 21
P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&	W 792086, M50001 W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	29.24 29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	26.42 26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	24.54 24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	22 23 23 27 20 26 26 24 21
P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&P&	W 792086, M60001 W 792086, M70001 W 792086, M80001 C 792009, 77B C 792009, 78B C 792009, 13C-2 W M50014A-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	29.38 30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	26.63 27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	24.60 25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	23 23 27 20 26 26 24 21
P& P& GE GE P& P& PO PO PO PO GE GE	W 792086, M70001 W 792086, M80001 CC 792009, 77B CC 792009, 78B CC 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSF-D-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	30.21 frozen 26.83 slushy frozen 31.98 27.30 37.32 27.97	27.36 31.21 24.05 29.89 29.37 28.92 24.74 24.74	25.36 29.11 22.03 28.00 27.76 26.68 22.85 22.84	23 27 20 26 26 24 21
P& GE GE P& P& PO PO PO GE GE DD	W 792086, M80001 CC 792009, 77B CC 792009, 78B CC 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D,	26.83 slushy frozen 31.98 27.30 37.32 27.97	24.05 29.89 29.37 28.92 24.74 24.74	22.03 28.00 27.76 26.68 22.85 22.84	20 26 26 24 21
GE GE P& P& PO PO PO GE GE	CC 792009, 78B CC 792009, 13C-2 CW M50014A-2 CW MJ0016A CW MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	slushy frozen 31.98 27.30 37.32 27.97	29.89 29.37 28.92 24.74 24.74	28.00 27.76 26.68 22.85 22.84	26 26 24 21
PS PS PS PO PO PO PO GE GE DD	CC 792009, 13C-2 W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	frozen 31.98 27.30 37.32 27.97	29.37 28.92 24.74 24.74	27.76 26.68 22.85 22.84	26 24 21
P&P&PO	W M50014A-2 W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	31.98 27.30 37.32 27.97	28.92 24.74 24.74	26.68 22.85 22.84	24 21
P& PO PO PO GE GE DD	W MJ0016A W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	27.30 37.32 27.97	24.74 24.74	22.85 22.84	21
P&PO PO PO GE GE DD	W MJ0013B OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	37.32 27.97	24.74	22.84	
PO PO PO PO GE GE DD	OSF-D-81-043, GECF-1D, JP-4 OSFD-81-044, GECF-13D, DF-2 OSF-D-81-042, GECF-14D, DF-2/ARO	27.97			71
PO PO PO GE GE DD	DF-2 DSF-D-81-042, GECF-14D, DF-2/ARO	frozan		25.00	21 21
PO PO GE GE DD	DF-2/ARO	1102011	30.54	28.58	27
PO PO GE GE DD	•				
PO GE GE DD		frozen	31.46	29.35	27
GE GE DD	OSF-D-81-046, GECF-1E, JP-4	27.66	25.03	23.11	21
GE DD	OSF-D-81-045, GECF-13E, DF-2 CCS-24D, Reference JP-4	frozen 27.53	30.50 24.78	28.54 22.75	26 21
DD	CCS-24D, Reference JP-4	27.63	24.89		21
	P-81-08 Blending JP-5,	27.00	21.07	22.00	
	Tank F-1, 4-23-81	30.17	28.55	26.63	25
	CCS-81B-1, DF-2	Crozen	30.07		26
GE	CCS-82B, DF-2	frozen	29.73	27.73	26
	SF-D-81-59, Blend of JP-4,				
	DF-2, 2040 Solvent	30.09	27.53	25.68	24
	PP-81-28, Tank F-12, AEDC		05 07	22 52	22
	Blend 2 (6 July 81) P-81-29, Tank F-11, AEDC		25.37	23.52	22
	Blend 3 (6 July 81)		27.46	25.60	24
	PP-81-30, Tank F-7, AEDC	-	2	20.00	₩.4
	Blend 5 (6 July 81)		29.66	27.53	25
DD	P-81-31, Tank F-9, AEDC				
	Blend 6 (6 July 81)		30.07	28.15	26
				(cont	inue
****		453			

TABLE 178 (continued)

	D	ynes per	centimeter	
Sample designation	-20°F ^a	32°F	70°F	100°F
E314A, JP-8 Blend #3 (P&W)	30.50	27.81	25.82	24.26
M50003B, JP-4 Blend #5 (P&W)	31.88	28.90	26.72	25.00
M50008B, JP-4 Blend #5 (P&W)	31.42	28.81	26.90	25.38
MS0007A, Shale JP-4 (P&W)	29.35	26.74	24.80	23.29
MS0010A, Shale JP-4 (P&W)	29.30	26.70	24.78	23.27
MS0011A, Shale JP-4 (P&W)	30.09	27.22	25.09	23.42
EJ0020B, JP-4 (P&W)	28.14	25.35	23.28	21.67
AEDC POSF-C-81-134, A-1 JP-4	28.75	25.95	23.85	22.27

aResults at this temperature were extrapolated from the higher temperature data.

TABLE 179. VAPOR PRESSURE VS. TEMPERATURE OF FUELS TESTED AFTER 1 MAY 80

		ters of m	
Sample designation	32°F	70°F	100°F
1B-792009, JP-4	37.5	85.0	149.0
2B-792009, JP-8	9.5	15.5	22.0
13B-792009, DF-2	9.5	14.5	19.0
14B-792009, DF-2 Aromatic blend	7.0	13.5	21.0
8B-792009	24.5	59.0	109.0
9B-792009	26.5	61.5	111.0
15B-792009	30.5	69.0	120.0
GEC-49B-1	33.5	78.0	141.0
1C-792009	35.0	81.0	145.0
8C-792009	23.5	58.0	108.0
9C-792009	29.5	71.0	131.0
13C-792009	7.0	12.0	17.0
14C-792009	6.5	11.5	17.0
15C-792009	29.5	63.0	106.0
JP-5, Tank 13	7.0	12.0	17.0
P&W ES0001, Shale JP-4	48.0	93.0	148.0
P&W 79-C-2086, Petroleum JP-4	28.5	71.0	136.0
P&W 79-C-2086, Shale JP-4	36.0	77.0	131.0

(continued)

TABLE 179 (continued)

Sample designation P&W 79-C-2086, Blend #1 P&W 79-C-2086, Blend #2 P&W 79-C-2086, Blend #3 P&W 79-C-2086, Blend #4 GEC 79-C-2009-62B-1, DF-2 P&W 792086, M50001 P&W 792086, M60001 P&W 792086, M70001 P&W 792086, M80001 GEC 792009, 77B	Millim	eters of m	mercury
Sample designation	32°F	70°F	100°F
P&W 79-C-2086, Blend #1	18.0	45.0	85.5
P&W 79-C-2086, Blend #2	13.5	34.0	66.0
P&W 79-C-2086, Blend #3	6.0	12.0	18.5
P&W 79-C-2086, Blend #4	53.0	120.0	212.0
GEC 79-C-2009-62B-1, DF-2	32.0	72.0	129.0
P&W 792086, M50001	47.0	94.0	152.C
P&W 792086, M60001	54.0	109.0	175.0
P&W 792086, M70001	8.0	15.5	24.5
P&W 792086, M80001	11.5	19.5	28.5
			123.0
GEC 792009, 78B	10.0	14.5	19.0
GEC 792009, 13C-2		12.5	16.0
P&W M50014A-2	6.0	11.5	18.5
P&W MJ0016A	30.0	73.0	137.5
P&W MJ0013B			140.0
POSF-D-81-043, GECF-1D, JP-4	32.5 32.0	75.0	133.0
POSF-D-81-044, GECF-13D, DF-2	7.0	12.0	16.5
POSF-D-81-042, GECF-14D, DF-2/AVO	8.0	13.5	
POSF-D-81-046, GECF-1E, JP-4	35.0		154.0
POSF-D-81-045, GECF-13E, DF-2	7.0	13.5	21.0
GECS-24D, Reference JP-4	34.5	79.0	139.0
GECS-26D, Reference JP-4	42.0	91.0	156.0
DDP-81-08 Biending JP-5, Tank F-1, 4-23-81	7.5	13.0	18.0
GECS-81B-1, DF-2	7.5	13.5	19.5
GECS-82B, DF-2	10.5	20.0	32.0
POSF-D-81-59, Blend of JP-4, DF-2,			
2040 Solvent	23.0	51.5	91.0
DDP-81-28, Tank F-12,			
AEDC Blend 2 (6 July 81)	24.5	60.0	110.0
DDP-81-29, Tank F-11,			
AEDC Blend 3 (6 July 81)	22.0	48.0	€3.0
DDP-81-30, Tank F-7,			
AEDC Blend 5 (6 July 81)	7.0	12.5	18.5
DDP-81-31, Tan: F-9,			
AEDC Blend 6 (6 July 81)	9.0	13.0	16.5
AEDC POSF-C-81-134, A-1 JP-4		62.0	

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TABLE 180. HEAT OF COMBUSTION OF FUELS TESTED AFTER 1 MAY 80

e a company and a company and a company and a company and a company and a company and a company and a company	 Gros	s values,	Btu/ib	Net Btu/lb	_
Sample designation	Duplic		Average	Average	<u>% на</u>
	20 106	20,097	20,102	18,783	14.46
1B-792009, JP-4	20,106		19,833	18,563	13.92
2B-792009, JP-8	19,839	19,826	19,523	18,322	13.16
13B-792009, DF-2	19,533	19,512	19,525	10,322	13.10
14B-792009, DF-2	10 144	10 005	10 120	18 033	11.92
Aromatic blend	19,144	19,095	19,120	18,033	12.08
8B-792009	19,234	19,243	19,239	18,137	13.14
98-792009	19,622	19,611	19,617	18,418	14.32
15B-792009	19,978	19,992	19,985	18,679 18,791	14.54
GEC-49B-1	20,141	20,093	20,117	18,694	14.38
10-792009	19,990	20,022	20,006	18,102	12.00
8C-792009	19,191	19,203	19,197		13.06
9C-792009	19,611	19,580	19,596	18,405 18,322	13.15
13C-792009	19,524	19,520	19,522	-	11.77
14C-792009	19,065	19,077	19,071	17,997	14.29
15C-792009	19,902	19,896	19,908	18,604	
JP-5, Tank 13	19,795	19,787	19,791	18,534	13.78 14.54
P&W 79-C-2086, Petroleum JP-4	20,022	19,974	19,998	18,672	
F&W 79-C-2086, Shale JP-4	20,035	19,993	20,014	18,701	14.39
P&W 79-C-2086, Blend #1	19,900	19,944	19,922	18,623	14.24
P&W 79-C-2086, Blend #2	19,708	19,743	19,726	18,500	13.44
P&W 79-C-2086, Blend #3	19,858	19,902	19,880	18,599	14.04
P&W 79-C-2086, Blend #4	19,454	19,404	19,429	18,310	12.27
GEC 79-C-2009-62B-1, DF-2	20,109	20,114	20,111	18,774	14.63
P&W 792086, M50001	19,704	19,738	19,721	18,409	14.38
P&W 792086, M60001	19,4€2	19,501	19,481	18,300	12.94
PSW 792086, M7C001	18,811	18,759	18,785	17,730	11.56
P&W 792086, M80001	18,857	18,906	18,382	17,833	11.50
GEC 792009, ?7B	20,033	19,995	20,014	18,695	14.46
GEC 792009, 78B	19,368	19,333	19,351	18,201	12.61
GEC 792009, 13C-2	19,496	19,498	19,497	18,305	13.07
P&W M50014A-2	19,667	19,627	19,647	18,421	13.44
P&W MJ0016A	20,115	20,118	20,117	18,793	14.51
P&W MJ0013B	20,094	20,140	20,117	18,796	14.48
POSF-D-81-043, GECF-1D, JP-4	20,060	20,073	20,067	18,735	14.54
POSF-D-81-044, GECF-13D, DF-2 POSF-D-81-042, GECF-14D,	19,193	19,204	19,199	18,100	12.04
DF-2/ARO	19,547	19,509	19,528	18,333	13.10
POSF-D-81-046, GECF-1E, JP-4	20,048	20,075	20,062	18,750	14.38
POSF-D-81-045, GECF-13E, DF-2	19,514	19,493	19,504	18,307	13.12
GECS-24-D, Reference JP-4	20,151	20,110	20,131	18,808	14.50
GECS-26-D, Reference JP-4	20,039	20,020	20,030	18,725	14.30
DDP-81-08 Blending JP-5	19,824	19,817	19,821	18,571	
GECS-81B-1, DF-2	19,582	19,560	19,571	18,391	
GECS-82B, DF-2	19,530	19,562	19,546	18,373	
POSF-D-81-59, Blend of JP-4,	25,000		,	•	
DF-2, 2040 Solvent	19,440	19,453	19,447	18,302	
DDP-81-28, Tank F-12, AEDC		,	_ ,	•	
blend 2 (6 July 81)	19,736	19,759	19,748	18,516	13.50
DDP-81-29, Tank F-11, AEDC	,	2.,	,	•	
blend 3 (6 July 81)	19,704	19,725	19,715	18,484	13.49
DDP-81-30, Tank F-7, AEDC	,,,	,,	,	,	
blend 5 (6 July 81)	19,329	19,362	19,346	18,206	12.50
DDP-81-31, Tank F-9, AEDC	27,523	20,002	27,519	,	
blend 6 (6 July 81)	19,612	19,632	19,622	18,391	13.49
AEDC POSF-C-81-134, A-1 JP-4	20,137	20,121	20,129	18,817	14.27
REPURIOR TO THE PROPERTY OF THE	20,107	20,121	20,225	_0,,	

aSupplied by AFWAL/POSF for net heat calculation.

(continued)

(FBP) 99.5 337 296 409 319 278 286 322 292 393 381 336 375 373 290 271 286 291 291 286 286 98 324 286 400 292 288 288 303 301 301 Temperatures at which the following percents were recovered (°C) 5 10 20 30 40 50 60 70 80 90 95 266 266 362 2887) 248 256 340 BY GC (ASTM D 223 246 318 202 237 302 174 229 287 °C, 142 220 271 SIMULATED DISTILLATIONS IN 118 215 255 169 219 217 218 206 195 269 250 250 181 251 251 251 251 251 140 103 206 240 89 196 222 219 219 199 106 105 133 99 98 156 129 236 227 227 145 195 162 198 198 198 197 119 153 184 184 184 182 74 181 196 204 199 199 186 87 178 102 65 58 168 177 170 170 167 167 88 184 174 94 187 3 25 138 141 150 152 35 35 151 151 137 140 127 120 129 144 (IBP) 0.5 21 125 121 130 139 129 104 98 103 131 21 77 74 TABLE 181 Shale JP-4 P&W 79-C-2086, Petroleum Sample Descriptions Blend #1 Blend #2 Blend #3 Blend #4 Paw ES0001, Shale JP-4 139-792009, DF-2 14B-792009, DF-2 Arometic blend JP-4 JP-8 P&W 79-C-2066, P&W 79-C-2086, 79-C-2086, P&W 79-C-2086, JP-5 2B-792009, 1B-792009, 15B-792009 13C-792009 14c-792009 150-792009 8B-792009 9B-792009 GEC-49B-1 LC-792009 8C-792009 90-792009 GEC-SPC2A GEC-S57A GEC-SICA GEC-S18A GEC-S21A GEC-S56A Fank 13, GEC-S3A GEC-58A GEC-S4A GEC-S9A

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TABLE 181 (continued)

	(IBP)		Temper	Temperatures		at which the		following	percer	percents were		recovered	(၃)		(FBP)
Sample Descriptions	0.5	-	2	10	20	30	40	20	09	70	96	90	95	66	99.5
SP 80-}	140	151	182	197	218	232	248	260	273	288	304	329	347	371	375
SP 80-2	145	157	188	204	226	241	255	269	283	299	315	340	360	385	391
SP 80-3	53	37	68	88	103	119	134	151	173	193	215	236	253	271	276
SP 80-4	93	111	162	178	195	205	216	222	231	240	250	263	270	279	280
	118	136	170	183	197	209	216	225	234	244	255	270	285	304	307
	116	133	169	184	197	508	218	228	236	246	251	272	285	301	305
	140	150	174	186	199	210	219	226	236	245	256	271	283	599	301
	142	151	178	190	204	216	226	236	244	259	275	303	323	350	354
	96	66	141	152	160	164	168	176	194	213	232	256	273	596	300
	38	23	5 8	106	135	151	191	163	168	172	189	221	247	282	289
SP 80-11	8	23	83	98	118	139	153	191	168	180	203	232	252	280	285
SP 80-12	¥	42	92	95	117	133	154	171	192	216	244	288	313	344	352
SF 80-13	146	159	186	199	218	234	247	259	272	787	304	329	349	375	379
BRF-9	9	2	69	88	101	120	143	167	189	210	230	253	273	302	307
B?F~13	138	151	181	199	221	241	256	271	287	304	319	344	363	392	397
BRF-14	140	154	182	196	218	230	246	258	273	291	312	338	360	396	404
CRF-8	38	20	62	82	142	179	195	211	225	232	247	263	278	302	306
CRF-13	147	797	193	211	234	253	268	282	295	308	324	347	366	39.1	399
14	150	164	150	203	225	234	254	7 97	276	291	308	328	343	362	364
79-c-200	5 6	67	99	88	100	118	128	141	152	162	174	190	5 06	253	265
792086,	ıc	27	89	111	136	156	174	186	198	213	225	248	270	309	314
792085, 1	10	27	63	83	120	141	172	194	216	234	256	289	320	353	376
792086, 1	132	134	137	140	159	166	186	217	236	276	304	326	3	349	253
792036, 1	145	157	167	187	206	224	237	251	264	280	298	329	347	373	379
792039,	92	35	61	73	95	117	137	158	175	193	210	233	251	289	311
792009,	122	142	184	201	221	236	250	261	271	285	301	323	343	391	405
GEC 792309, 13C-2	103	123	179	200	225	244	258	271	287	302	316	341	357	388	397
P&W M5G014A-2	112	115	139	161	184	197	208	217	224	235	244	362	288	348	365
PSW MJ0016A	27	35	53	29	93	117	141	172	196	213	228	244	254	274	285
P&W MJ00133	62	37	28	69	6	117	152	179	197	215	223	5¢6	256	285	308
	5 8	34	67	87	100	118	132	151	172	194	215	237	253	285	30.5
	133	147	178	195	219	237	252	267	281	298	315	338	355	387	398
FOSE-U-05: U4Z, GECE-14U,		;	,		į	9	;		,	1	į	•	;		
DF-Z/ARO	138	151	9 8	195	215	877	241	253	592	282	304	329	349	383	394

TABLE 181 (continued)

(FBP)	99.5	304	399	303	}	356	323	309		307	403	402		383		300	306	302	315	309	441	421	444	312	371	215		412	316		415	318
	66	283	390	277	:	384	300	291		296	380	390		370		288	292	289	304	300	425	412	428	302	358	202	767	399	301		405	772
(°C)	95	256	355	235	}	348	257	271		273	356	355	ı	330		26.0	260	260	274	272	385	381	385	273	286	25.4	1	357	569		367	254
Temperatures at which the following percents were recovered (°C)	8	248	339	218		330	243	247		264	338	337		307		246	247	247	263	2F 2	365	361	364	506	556	237	153	331	255		344	238
re re	8	233	318	195		305	221	226		252	316	314		277		228	225	227	249	248	340	327	339	187	247	215	717	367	238		315	217
nts ve	20	218	303	176)	288	201	208		241	298	297		254		208	208	208	237	236	320	318	319	185	233	107		267	227		292	196
perce	9	207	290	160		273	180	168		232	281	285		236		187	186	187	229	228	304	303	303	163	230	371	3	242	215		270	173
lowing	20	194	276	143		260	157	166		223	997	264		223		161	160	191	220	219	291	288	289	181	221	150		220	203		252	153
he fol	40	171	263	128		248	135	141		216	251	249		203		136	136	137	213	213	274	272	272	180	211	1 30	}	197	192		236	135
hich t	30	142	248	113		233	118	120		206	235	233		179		120	120	119	204	203	259	255	256	178	208	3118		163	171		224	117
s at	20	16	229	96		216	100	101		961	217	215		147		100	101	101	194	193	240	236	238	173	202	ő	•	132	162		208	86
rature	2	65	204	72		194	87	38		181	195	187		102		88	88	83	181	181	215	213	214	158	200	ä	3	98	159		192	82
Tempe	2	28	186	61		178	69	69		169	175	162		88		69	20	69	171	171	193	192	192	167	196	67	;	98	152	i	176	63
	-	42	134	36		146	40	41		142	125	100		58		38	39	40	150	150	150	150	147	158	184	<u>د</u> بر)	28	145	!	145	34
(IBP)	0.5	35	98	27		133	33	33		129	105	98		39		27	5 6	27	142	142	136	134	132	157	181	25	3	43	142		131	27
	Sample Descriptions	PGSF-D-81-046, GECF-1E, JP-4	POSF-D-81-045, GECF-13E, DF-2 DDP-81-25, Rlend JP-4	Tank B-1, 5 May 81	DDP-81-26, DF-2 Base Stock,	5 May 81	GECS-240, Reference JP-4	GECS-26D, Reference JP-4	DDP-81-08, Blending JP-5	Tank F-1, 23 April 81	GECS-81B-1, DF-2	GECS-82B, DF-2	POSF-D-81-59, Blend of JP-3,	DF-2, 2040 Solvent	AEDC Blending Stocks		DDP-81-23, Tank B-12, JP-4	, Tank B-13,	, Tank F-6,	, Tank F-7,		, Tank F-10,	, Tank F-11	, Tank F-8, X	Tank B-18,	DDF-81-28, Tank r-12, AEDC Rlend 2 (6 July 81)		\sim	(6 July 81)	DDP-81-31, Tank F-9, AEDC	Blend 6 (6 July 81) AEDC POSE-C-81-134 A-1	JP-4

(continued)

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(FBP) 99.5 714 557 534 554 572 572 555 556 565 768 590 574 622 572 572 572 730 730 563 595 558 750 718 637 707 707 703 520 550 550 563 547 752 566 532 547 Temperatures at which the following percents were recovered (°§) 5 10 20 30 40 50 60 70 80 90 95 684 522 469 498 538 678 489 520 93 509 460 505 9 **64**8 **6**30 93 2 **50**7 60 475 604 ಜ್ಞ 459 576 130 153 586 586 563 426 552 552 552 471 \$60 532 406 529 527 527 527 453 450 444 549 356 388 428 520 88.5 137 419 491 410 248 403 327 403 464 406 408 306 372 489 450 457 335 432 208 331 293 26 \$26 390 223 383 387 383 273 250 250 307 352 201 157 399 246 390 367 189 352 307 363 360 356 221 187 198 358 385 217 183 154 194 334 351 338 333 180 149 147 136 153 **4** 291 77 106 127 302 306 95 95 198 198 261 248 264 280 286 72 55 55 127 127 241 30 (IBP) 0.5 9.8 255 57 582 282 282 268 257 250 79-C-2086, Petroleca JP-4 Shale JP-4 Sample Descriptions Blend #2 Blend #3 Blend #4 P&W ES0001, Shale JP-4 Blend DE-2 DF-2 Aromatic blend JF-4 JP-8 79-C-2086, P&W 79-C-2086, 79-C-2086, 79-C-2086, P&W 79-C-2086, Tank 13, JP-5 13B-792009, 14B-792009 13C-792009 14c-792009 15B-792009 15c-792009 1B-792009, 2B-792009, 88-792009 9B-792009 IC-792009 8c-792009 9c-792009 GEC-49B-1 GEC-SPC2A GEC-S) 8A GEC-S21A GEC-S10A GEC-S56A GEC-S57A GEC-S3A GEC-S8A GEC-S4A GEC-S9A P&W P6W

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GC (ASTM D 2887)

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SIMULATED DISTILLATIONS IN

TAPLE 182.

TABLE 182 (continued)

•	(1BP)		Tempe	Temperatures		iich tl	at Which the following	loving	Dercente cere	, ee .	•	6461	(40/		(444)
Sample Descriptions	0.5	7	2	10		30	4	S	9	70	90	80 90	2 6	8	
SP 80-1	284	706	000	ç											
SP 80-2	200	7 7	ָ מַנְּיִלְ מַנְיִילְ	200	676	450	478	200	2 53	2 20	579	624	657	00,	707
SP 80-3		010	2 ;	7	433	400	491	216	Z	570	299	644	9	727	736
	\$ 5	ָרָ ק	154	061	217	246	273	304	343	379	419	457	487	520	529
	133	727	324	352	383	401	421	432	448	494	482	502	518	534	536
	767	117	338	361	387	408	421	437	453	471	491	518	545	579	7.87
	241	271	336	363	387	408	424	445	457	475	484	522	25	574	£ 5
	284	302	345	367	390	410	426	439	457	473	493	520	3		274
	288	304	352	374	399	421	439	457	471	498	527	27.7	;		
	187	210	286	306	320	327	334	249	38.	£15	3 5	707	210	70	8 6
	601	135	192	223	275	304	322	325	334	CYE	200	100	223	6	5/2
	104	135	181	208	A4.C	250	2 6	666	7	3 2	7/0	674	1/6	ž	255
SP 80-12	ð	108	160	201	27.2	32.5	3 6	775	400	326	397	450	4	236	2 5
SP 80-13	20,0	3 6	207	170	C#7	7/7	ָרָהָ מַרְהָי	Š	378	421	471	550	595	651	999
G-Laga	627	010	9	3	676	453	477	498	522	5 49	579	624	99	707	714
	4.	104	156	192	214	248	289	333	372	410	6.5	4.87	523	576	Š
CT - VG	280	304	358	390	4 30	466	493	520	549	579	909	651	583	737	747
DATE TO SERVICE	284	309	360	382	424	4 46	475	496	523	556	594	640	989	745	750
	100	122	144	180	288	354	383	412	437	450	477	50.5	23	46	, CB 7
	297	324	379	412	453	487	514	540	563	586	615	657	169		750
14	302	327	374	397	437	453	482	503	529	556	586	622	7 0 9 9	765	700
/9-C-200	79	\$	151	1.90	212	244	262	286	306	124	345	374	}		6
792086,	41	81	190	232	277	313	345	367	388	415	4 37	47.4	2 2	0	110
792086,	20	81	145	192	248	286	347	381	421	452	40.4		010	8 5	160
792086, 1	270	273	279	284	318	331	367	423	457	524	£73	200	909	8 9	5 5
792086,	293	315	333	358	403	435	459	484	507	53.6	26.2	624	5 2 2	3 6) ;
	79	95	142	163	203	243	279	316	247	320	5 5	770		3 5	617
	252	288	363	394	430	4	482	503	, C	272	015	10#	\$;	766	269
GEC 792009, 13C-2	217	253	354	392	437	471	496	200	5.49	27.5	F/7	545	40	9 6	19/
P&W M50014A-2	234	239	282	322	363	387	406	423	436	726	3 5) t	ה ל ה	3 (141
P&W MJ0016h	81	95	136	153	100	242	200	770	70	2 .	7/4	# OC	000	929	689
P&W MJ0013B	84	66	136	156	194	243	306	240	200	410	7447	4/1	489	525	545
POSF-D-81-043, GECF-1D, JP-4	79	6	153	081	212	244		֓֞֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓	700	614	754	4 / D	493	2	586
ے .	27.1	20,0	25.5	101	717	54.7	2/7	304	342	381	419	459	487	54 5	581
, GECF-14D	;	7	700	700	97	40.4	486	513	538	268	299	64 0	671	729	748
DF-2/ARO	280	304	356	383	419	442	466	487	516	545	570	773			;
				, ,	ı J	•	<u>}</u>	-	2	ŗ	7.70	4 70	200	17/	/41

TABLE 182 (continued)

	(IBP)	j	Temper	Temperatures		at which the following	follo		percents were	ts ver		recovered	(°F)		(FBP)
Sample Descriptions	0.5	1	5	01	20	30	40	20	09	70	80	90	95	8	99.5
A_GT	Q	90	136	971	106	000	126	6	304	724	154	470	402	5	679
1035-D-01-046, GECF-1E, JF-4 BO:1-0-01-046, GECF-126, DE-2	600	333	252	200	277	770	100	100		177	107	275		726	750
r.sr-p-81-043, GECF-135, DF-2 DDP-81-25, Blend JP-4,		7	Š				3	676	ţ	5	5	5		Ş	3
Tank B-1, 5 May 81	81	6	141	161	205	235	262	239	321	349	382	425	455	531	578
DDP-81-26, DF-2 Base Stock,															
5 May 81	271	295	352	381	420	451	478	200	523	551	285	62 5	629	723	74
GECS-24D, Reference JP-4	92	105	157	189	211	244	275	315	357	395	430	469	4 8	572	613
GECS-26D, Reference JP-4	35	105	156	191	213	248	285	330	371	407	440	476	519	555	588
DDP-81-08, Blending JP-5,															
Tank F-1, 4-23-81	264	288	336	. 58	385	403	45,	433	450	4 66	486	207	523	%	58 3
GECS-81B-1, DF-2	221	257	347	383	423	455	484	511	538	268	601	640	673	734	757
GECS-82B, DF-2	187	212	324	369	419	451	480	507	536	267	283	639	671	734	756
POSF-D-81-59, Blend of JP-4,															
DF-2, 2040 Solvent	102	136	190	221	297	354	397	433	457	489	531	585	979	869	721
AEDC Blending Stocks															
DDP-81-22, Tank B-11, JP-4	81	100	156	192	212	248	277	322	369	406	439	475	200	550	572
DDP-81-23, Tank B-12, JP-4	79	102	158	192	214	248	277	320	367	4 0 6	437	477	30S	558	583
	81	104	156	191	214	246	279	322	369	4 0 4	ŧ	477	200	552	576
DDP-81-20, Tank F-6, JP-5	288	302	339		381	399	415	428	4	458	48 0	505	525	579	2 88
	288	302	339	358	380	398	415	427	4 3	457	479	,03	522	573	635
, Tank F-9,	276	303	380	419	465	498	2 76	555	579	609	643	688	725	197	825
DDP-81-18, Tank F-10, DF-2	274	301	378	416	458	492	521	551	515	605	639	682	717	773	789
DDP-81-19, Tank F-11, DF-2	569	297	377	417	460	493	522	552	578	607	641	688	726	803	831
DDP-81-12, Tank F-8, Xy-B	315	216	333	334	343	352	356	358	361	365	369	403	523	\$ 76	594
Tank B-18,	358	363	385	392	401	406	412	4 30	44 6	451	477	493	74	9/9	200
CDP-81-28, Tank F-12, AEDC															
(6 July 81)	11	95	153	187	208	244	.42	318	334	387	419	459	489	558	299
DDP-81-29, Tank F-11, AEDC						,		ı	,		1	1	!	1	,
(e July 81)	109	136	187	208	270	325	387	428	468	51 3	26 5	628	675	750	774
DDP-81-30, Tank F-7, AEDC					;	:	į		1	,	;	;	;	į	į
$\overline{}$	288	293	306	318	324	340	378	397	419	141	460	491	515	5/4	109
DDP-31-31, Tank F-9, AEDC		,	;		;	;	ļ	į	;	1	6	į	6	,	1
Blend 6 (6 July 81)	3 2 8	293	349	378	406	435	457	486	518	558	546	159	693	19/	61.
AEDC POSF-C-81-134, A-1,	ă	25	145	081	800	243	275	307	£ 9E	385	423	460	489	531	504
# .uc	5	, י	7	3	2	7	1	<u>}</u>	?)	}) })	1	;

(continued)

Sample designation	Daraffine	Monocyclo-	Dicyclo-	Alkyl	Indans/	:
11013 5251 535	CILL AL ALL	Par attills	pararrus	Denzenes	tetralins	Naphthalenes
GEC-120-4A0-792033	37.3	23.0	0.0	12.8	4	\$ C2
GEC-120-8A0-792033	38.9	20.1	0.0	•	•	24.1
JP-8/Aro-12(772043)	30.7	28.0	2.5	11.2		21.5
JP-4/Aro-12(772043)	32.2	7.72	1.6		, ru	21.7
1B-792009, JP-4	68.8	15.3	2.4			. 0
•	43.6	39.1	3.7		4.1	6.7
	45.8	32.5	2.6	8.4	, v.	4.2
14B-792005, DF-2 Aromatic blend	35.0	24.5	2.0	11.1	6.5	6.07
8B-792069	42.5	16.2	0.0	14.8	4.4	22.1
9B-792009	46.0	27.1	0.0	12.4	5.6	11.9
15B-7920c3	45.6	43.4	0.0	7.4		0.0
GEC-49B-1	58.5	27.9	2.8	9.1		0.7
Reanalyses of blends and stocks						
1A	61.4	23.6	5.0	8.5	1.0	0.5
2 A	43.2	39.9	3.7	7.4	3.9	1.9
38	44.0	39.7	3.5	7.2	3.8	1.8
48	30.6	27.9	2.6	11.1	5.8	22.0
5.A	34.4	31.0	3.3	28.0	1.7	1.6
6A	25.4	20.8	1.6	51.4	0.0	8.0
₩	36.8	34.0	3.2	9.3	4.8	11.9
2	36.1	22.2	0.0	12.9	4.3	24.5
		27.4	0.0	11.3	2.7	13.7
10%	31.7	12.4	•	52.7	0.4	4.0
		17.2	•	34.3	9.0	0.4
1.28	57.2	22.2	5.2	13.8	1.1	
13A-1	45.7	30.4	•	8.1	6.0	7.5
	•	32.5	3.2	7.6	4.8	6.5
Xylene bottoms	1.2	0.1	0.0	98.7	0.0	0.0

TABLE 183 (continued)

Sample designation	Paraffins	Monocyclo- paraffins	Dicyclo- paraffins	Alkyl benzenes	Indans/ tetralins	Naphthalenes
GMSO	58.2	35.0	0.0	3.5	2.5	0.8
2040 so?vent	3.6	6.7	0.1	19.4	9.8	4.39
1C-792009	58.5	21.1	7.7	9.0	2.5	1.2
8C-792009	36.1	19.8	0.0	13.2	5.6	25.3
9C-792009	9:21	24.9	0.0	11.6	4.2	13.7
13C-792009	46.1	31.3	2.4	8.0	7.4	4.8
14C-792609	32.4	21.6	1.7	11.6	8.2	24.3
15C-792009	43.7	45.7	0.0	6.4	3.2	1.0
772043-F1	62.3	21.8	5.5	8.4	1.,	0.7
772043-F2	44.1	39.3	3.6	8.0	3.1	1.9
772043-F3	45.1	39.9	2.5	7.6	3.1	1.8
772043-F4	31.1	27.0	1.9	11.6	5.4	23.0
772043-F5	35.1	30.7	2.9	28.6	6.0	 8. T
772043-F6	25.3	20.8	1.9	51.1	0.0	6.0
772043-F7	37.6	33.1	2.2	6.6	4.3	12.9
772043-F8	36.6	20.8	0.0	12.9	4.7	25.0
772043-F9	46.0	25.8	0.0	11.3	3.3	13.6
772043-F10	31.6	11.4	2.6	53.9	0.0	0.5
772043-F11	44.4	15.9	3.7	34.8	9.0	9 0
77-043-F12	57.5	20.9	5.4	14.3	1.2	0.7
77-043-73	44.3	32.2	2.6	8.6	6.3	0.9
JP-5, Tank 13	43.4	35.1	4.1	10.2	4.4	2.8
P&M ES0001, Shale JP-4	44.7	45.7	0.0	6.4	3.2	0.0
P&W 79-C-2086, Petroleum JP-4	62.6	20.9	5.4	9.5	1.7	0.2
P&W 79-C-2086, Shale JP-4	44.3	45.6	0.0	9.9	3.5	0.0
P&W 79-C-2086, Blend #1	58.1	31.4	0.0	5.9	2.5	2.1
	46.0	26.2	0.3	25.4	1.4	0.7
P&W 79-C-2086, Blend #3	49.2	33.1	3.9	9.7	₽.G	0.7
P&W 79-C-2036, Blend #4	40.3	4.2	0.1	52.9	2.1	4.0
GEC 79-C-2009-62B-1, DF-2	51.3	37.5	3.5	6.9	0.3	0.5

(continued

(continued)

	TAI	BLE 183	(continued)			,
Sample designation	Paraffins	Monocyclo- paraffins	Dicyclo- paraffins	Alkyl	Indans/ tetralins	Naphthalenes
PSW 792086 M50001	45.1	45.2	0.0	4	et et	0.0
792086,	46.4	8	0.2		•	3.8
	17.5	16.4	4.5	56.5	1.1	4.0
P&W 792086, M80001	33.5	18.7	2.3	17.6	13.0	14.9
GEC 792009, 77B	63.6	19.5	4.3	11.2	1.2	0.2
GEC 792009, 78B	42.0	30.6	3.3	7.7	9.0	10.4
GEC 792009, 13C-2	44.1	31.3	2.9	8.1	7.4	5.7
M50014A-	48.1	28.8	3.0	13.6	3.7	2.8
P&W MJ0016A	64.4	19.1	5.9	7.9	2.0	0.7
PGW MJ0C13B	63.5	19.3	6.4	8.0	2.0	0.8
	62.0	23.6	4.2	8.4	1.7	0.1
	35.0	25.4	1.9	10.9	6.7	19.6
POSF-U-81-042, GECF-14D,					,	
	45.4	32.3	2.4	8.5	ហ	5.9
GECF-IE, J	60.1	21.0	7.0	8.4	1.7	0.1
POSF-D-81-045, GECF-13F, DF-2	47.3	32.2	1.9	7.9	6.4	4.3
DDP-81-25 Blending JP-4,						
Tank 8-1, 5-5-81	26.0	29.3	2.7	10.1	1.0	6.0
DDP-81-26 DF-2 Base stock,						
5-5-81	41.9	32.2	3.1	8.3	6.3	8.2
GECS-24-D, Reference JP-4	63.2	22.2	3.7	9.1	1.7	0.1
GECS-26-D, Reference JP-4	61.5	20.4	4.1	12.0	1.8	0.2
	45.4	38.9	2.8	7.5	3.0	2.4
Tank F-1, 4-23-81						
GECS-81B-1, DF-2	44.9	32.6	2.2	8.7	5.8	2.8
•	45.7	32.9	1.7	8.7	5.5	5.5
1-59						,
DF-2, 2040 Solvent	40.5	34.2	0.0	ر د	ď	م د

TABLE 183 (continued)

Sample designation	Paraffins	Monocyclo- paraffins	Dicyclo- paraffins	Alkyl benzenes	Indans/ tetral:ns	Naphtha cors
AEDC Blending Stocks						
DDP-81-22, Tank B-11, JP-4	65. i	19.1	3.6	10.3	1.8	0.1
	65.2	19.0	3.6	10.3	1.8	0.1
Tank B-13,	65.2	18.9	3.6	10.4	1.8	0.1
, Tank F-6,	45.0	35.0	3.1	8.5	6.1	1.9
, Tank F-7,	6.4	34.9	3.2	8.9	6.2	1.9
DDP-81-17, Tank F-9, DF-2	45.7	38.6	2.0	6.2	4.4	3.1
, Tank F-10,	45.7	39.2	1.7	6.1	4.3	3.0
	45.6	38.6	2.0	6.2	4.5	3.1
F-8,	1.2	1.0	0.0	7.96	9.0	0.5
	2.5	9.0	0.1	22.4	23.3	51.1
Blend 2 (6 July 81)	54.0	19.6	0.3	19.7	2.7	3.7
DDP-81-29, Tank F-11, AEDC						
(6 July 81)	48.8	30.6	0.0	13.2	3.6	3.8
DDP-81-30, Tank F-7, AEDC						
Blend 5 (6 July 81)	33.0	24.6	2.3	31.5	4.4	4.2
DDP-81-31, Tank F-9, AEDC						
Blend 6 (6 July 81)	44.7	36.2	2.1	7.6	5.4	4.0
E314A, JP-8 Blend #3 (P6W)	49.7	33.3	2.9	10.3	3.2	9.0
M50003P, JP-4 Blend #5 (P6W)	48.1	29.0	2.5	14.7	3.6	3.1
M50008B, J7-4 Blend #5 (P&W)	47.1	29.2	2.2	15.0	3.5	3.0
MS0007A, Shale JP-4 (P&W)	44.1	45.5	0.0	6.7	3.6	0.1
MS0010A, Shale JP-4 (P&W)	44.2	45.6	0.0	6.9	3.2	0.1
Shale JP-4	43.8	45.9	0.0	6.8	3.4	0.1
JP-4 (P&W)	56.3	25.8	4.0	13.0	0.5	4.0
AEDC POSF-C-81-134, A-1 JP-4	59.8	21.6	4.5	12.3	5.1	0.3

COMPARATIVE HYDROCARBON-TYPE ANALYSES BY MODIFIED ASTM D 2789 AND MONSANTO METHOD 21-PQ-38-63, IN WEICHT PERCENTS TABLE 184.

								CINDUNG TOUTH HEREENTO	ロシノロコ	n		
	GEC-120-	GEC-120-7-3-792033	GEC-120-880-792033	0-792033	. P-6/Aro-12(772043)	12(772043)	JP-4/Aro-12(772043)	2(772043)	28-792009, JP-8	JP-8	138-742004 pg-2	
Squarodino	D 2789	MONS	D 2789	MONS	F. 2769	HOMS	D 2789	HOMS	D 27.80	1	7777	
Paraffins	33.3	29.7	3	ş	;						0 6/63	
Monocycloparaffins	22.1	17.1	19.2	15.0	26.8	2.07 2.02	28.6	25.8	41.0	40.2	42.7	12.0
Dicycloparaffins	0.0	u,	0.0		2.4	f. 6.	9.9	₹. 92	79.7	43.6	32.8	33.8
Thing thensenes	13.3	18.3	13.1	17.9	11.5	14.4	12.0	. 41	10 , 0	, (
Independ to	4.1	9.6	4.7	4.1	6.2	4.8	9.6	7 7	7.	, .		9
March the Length 10	, t	0.5	ı	0.5	•	9.0			•	÷ ;		+ .
	2.12	30.5	28.5	31.6	25.9	25.6	25.7	28.5	7 •	0 0 4	•	0,6
	148-792000 ng-3	0 56-7							;	?		6. 1
÷	Aromatic Blend	Blend	88-792009	6003	98.79 2009	2000	3					
Components	D 2789	HOMS	D 2789	MOMS	2780		60076/ -BCT	600	Blend 4	3	Blend 7	
•					2 2103	MARS	68/2 a	HORES	D 2789	HORS	D 2789 IK	12
Paraffins	31.1	30.5	37.9	34.5	107	,	;	,				İ
nonocycloparafrins	23.5	24.3	15.6	9.6	25		43.2	41.4	27.1	26.2		2.9
Dicyclipperacting	1.9	1	0.0		0		•	5.	7.92	78.7		36.5
ALKYL COURSE	11.5	13.6	15.3	21.5	13.2	- 6		٠;	2.5			_
Thomas of the state of the stat	7.2	5.5	4.9	₩.¥	3.0		7 7	10.8 2.0	11.4 0.7	14.1	9.9	11.9
Manager Shift 10	ı	1.3	•	0.5	: •) -	7 .		6 .9	V		+.+
Maphiculteness	24.8	24.8	26.3	29.6	14.6	17.3		9 (• ;	5,5		7.0
						?		D. D	26.3	25.5		1.2

TABLE 184 (continued)

	Blen	lend 8A	Blend 9A	46	Blend 13A-1	134-1	Blend 13A-2	138-2	GNSO Stock	tock	2040 Selvert	Liverit
Components	D 2789	NOMS	D 2789	HOMS	D 2769	HOMS	D 2789	HOMS	D 2789	HOMS	0 2789	
Paraffins	31.9	28.4	41.1	38.5	42.4	41.3	42.3	41.3	55.8	\$6.3	2.8	1.5
Monocycloparaffins	21.2	16.9	27.1	27.7	30.4	31.1	32.7	3	36.3	35.0	9.0	•
Dicycloparaffins	0.0	۱,	0.0	•	2.3	1	3.2	,	0.0	1	6.1	•
Alkylbenzenes	13.3	18.0	12.0	16.7	8.7	10.2	8.2	9.1	3.9	4.2	17.6	22.1
Indans/Tetralins	4.7	4.1	3.1	2.9	6.9	5.6	N,	4.5	3.0	2.5	9.5	6.9
Indenes/C.H2,-10	•	9.0	•	0.3	1	0.7	•	1.0	,	0.7	•	1.2
Maphthalefie."	58.9	32.1	16.7	19.4	9.3	11.3	8.1	4 ,	1.0	1.3	7.5	67.9
				;							PO6F-C-61-13	11-134
	SC-79	6002	90-792009	600	13C-792009	2003	14c-792009	5003	15c-792009	2003	MEC 1-1 31-	7-45 1
Components	D 2789 NO	HOMS	D 2789	SHOW.	0 2789	HOHE	D 2789	SHOP	D 2789	2002	0 200	SHORE
Paraffins	31.8	27.3	41.6	37.6	42.9	42.4	28.5	28.1	41.3	0.0	57.2	51.6
Monocycloparaffins	18.9	16.0	24.6	21.7	31.5	32.2	20.7	21.7	4.6	\$6.5	22.3	X
Dicycloperaffins	0.0	•	0.0		2.4	1	1.6	•	0.0	,	4.7	•
Alkylbenzenes	13.5	18.4	12.3	17.3	6.7	10.0	3.1.8	14.2	7.0	9.0	13.6	S.
Indans/Tetralins	6.1	5.2	4.8	+.4	8.5	9 .9	6.9	6.8	3.8	3.3	1.0	1.9
Indenes/C_H210	•	0.5	•	0.2	•	1.0	•	1.2	•	0.0	•	0.0
Maphthalenes"	7.62	32.6	16.7	18.5	6 .0	7.6	28.5	28.5	1.3	1.4	• .	0.1

TABLE 184 (continued)

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B nd #5	MONS	42.3	30.4	. ,	18.8	3.7	0.0	4 .8	•	1	MONS		34	í ,	21.3	0	0.0	0.0
M50003B JP-4 Blend #5	D 2789	44.7	29.1	2.5	15.8	4.1		3.8	E.10020B	JP-4	D 2789	53.8	26.6	4.1	14.4	9 0		0.5
A end #3	HONS	45.0	47.4	•	13.8	3.1	0.0	0.7	1.8	JP-4	MONS	40.9	45.8		9.5	3,5	0.0	0.3
E214A JP-8 Blend #3	D 2789	47.1	34.1	3.0	11.2	3.8	•	8.0	H50011A	Shale JP-4	D 2789	41.5	46.9	0.0	7.5	4.0	ı	0.1
Fank F-9 5(7-6-81)	NONS	41.0	37.9		9.6	5.0	9.0	5.6	¥.	IP-4	HONS	40.7	45.9		6.6	3.3	0.0	0.2
DDP-81-31, Tank F-9 AEDC Blend 6(7-6-81)	D 2789	41.9	36.6	2.1	8.2	6.2	•	5.0	MS0010A	Shale JP-4	D 2789	41.9	46.6	0.0	7.6	3.8	•	0.1
Tank F-7 5(7-6-81)	MONS	27.7	24.7	•	38.0	4.5	. 0.0	5.1	7.A	JP-4	r. JAS	41.3	45.8	•	9.6	3.4	0.0	0.2
DDP-81-30, Tank F-7 AEDC Blend 5(7-6-81	n 2789	30.1	24.2	2.3	33.3	5.0	•	5.1	M50007A	Shale JP-4	D 2789	41.8	46.5	0.0	7.4	4.2	•	0.1
Tank F-11 3(7-6-81)	MONS	43.6	28.3	,	18.3	3.9	0.3	5.6	ĸç	nd #5	MONS	42.2	31.0	•	18.5	3.7	0.0	5.5
DDP-81-29, Tank F-11	D 2789	45.7	31.0	0.0	11.4	4.1	•	4 .8	#50003#	JP-4 Blend #5	D 2789	0.44	29.6	2.3	16.3	4.1	•	3.7
	Components	Paraffins	Monocycloparaffins	Dicycloparaffins	Alkylbenzenes	Indans/Tetralins	Indenes/CH2, 10	Naphthalenes"		ı	Components	Paraffins	Monocyclopareffins	Dicycloparaffins	Alkylbenzenes	Indans/Tetraling	Indenes/C _N H ₂ ,-10	Naphthalenes"

^aModified ASTM Method D 2789.

b Honsanto Method 21-PQ-38-63, which combines mono- and di-cycloparaffins into a single value.

 $^{\sf C}_{\sf A}$ dash indicates the compound is not detected by the method.

TABLE 185. COMPARATIVE HYDROCARBON-TYPE ANALYSES WITH THREE MASS SPECTRAL METHODS INCLUDING ASTM D 2425, IN WEIGHT PERCENTS

		F-D-81-04 F-13D, DF	_		5F-D-81-04 14D, DF-2/			81-17 9, DF-2
Components	D 2789ª	D 2425 ^b	MONSC	D 2789	D 2425	MONS	D 2789	D 2425
Paraffins	42.3	40.6	41.1	31.7	32.7	30.8	43.0	40.2
Total cycloparaffins:	34.9	29.3	33.2 _d	26.3	22.9	24.7	41.2	36.8
Mono-	32.5	16.7	_a	24.5	13.3	-	39.2	25.2
Di-	2.4	9.0	-	1.8	7.0	•	2.0	9.0
Tri-	-	3.4	-	-	2.6	-	-	2.6
Alkylbenzenes	9.2	9.4	10.4	11.3	12.0	13.4	6.8	7.3
Indans/Tetralins	6.3	5.3	5.2	7.4	5.7	5.8	5.1	4.7
Indenes/C.H210	-	1.9	1.1	-	2.2	1.2	-	1.9
Naphthalehe n	•	0.4	-	-	1.8	-	-	ა.3
Total Naphthalenes	7.3	8.6	9.0	23.3	21.2	24.1	3.9	5.0
Acenaphthenes	•	2.6	-	-	1.9	-	-	2.0
Acenaphthylenes	_	1.6	-	-	0.5	-	-	1.2
Tricyclic aromatics	•	0.5	-	-	0.9	-	-	0.9
		SF-D-81-04 F-13E, DF-			-14, Tank		DDP-81-12 Xylene	, Tank F-8 Bottoms
Components	D 2789	D 2425	MONS		D 2425 ^f	MONS	D 2789	D 2425 ^f
Paraffins	44.2	41.1	43.3	2.0	0.0	0.8	1.1	0.0
Total cycloparaffins:	34.4	27.5	32.6	0.6	0.4	0.0	0.9	0.0
Mono-	32.5	16.0	-	0.5	-		0.9	-
Di-	1.9	8.5	-	0.1	-	-	0.0	_
Tri-	_	3.0	-	-	-	-	-	-
Alkylbenzenes	8.6	10.2	9.9	20.6	26.8	29.3	96.8	97.3
Indans/Tetralins	7.5	6.9	6.0	22.9	16.4	17.2	0.6	0.0
Indenes/CyH2n-10	-	1.9	1.1	-	0.0	0.6	-	0.0
Naphthalehe n	-	0.4	-	-	21.0	_	-	0.0
Total Naphthalenes	5.3	7.4	7.1	53.9	52.8	52.1	0.6	1.0
Acenaphthenes	•	2.6		•	1.9	_		1.3
Acenaphthylenes	_	1.5	-	•	1.1	-	-	0.2

^aModified ASTM Method D 2789; data determined in volume percents and converted to weight percents with average density values.

basTM Method D 2425 is preceded by an ASTM D 2549 fractionation whereby fuels are separated into saturate and aromatic fractions.

 $^{^{\}mathbf{C}}$ Monsanto mass spectral Method 21-PQ-38-63, which was developed for hydrocarbon feed stocks.

dA dash in a column indicates the compound can not be detected by the method.

e Includes naphthalene

There was no ASTM D 2549 fractionation for these fuels since they were essentially all aromatic. The D 2425 analyses were conflucted on the neat fuels which were considered as aromatic fractions for the calculations.

SECTION V

HIGH DENSITY FUELS

Certain advanced Air Force strategic missiles require high volumetric energy fuels because of the limited space available for the fuel. Several high density fuels have been developed based on synthetic cyclic hydrocarbons. These fuels, which include RJ-4, RJ-5, RJ-6, JP-9, and JP-10, consist of a relatively small number of chemical compounds and, thus, can be characterized by chemical composition as well as by bulk properties. The tasks described in this section pertain to defining the composition and properties of these high density fuels for engineering applications and quality control purposes, or are related to solving specific contamination problems that occur with the use of these fuels.

1. IDENTIFICATION OF CONTAMINANTS IN JP-9 TEST FUELS

JP-9 fuel specimens, which were used by an Air Force contractor in studies of fuel/elastomer compatibility, were examined to determine the kinds and amounts of contaminants present in the fuels. Four test fuels and one control fuel were involved in the evaluation. The purpose of the study was to determine whether the contamination of the fuel was due to components extracted from the elastomers, or due to oxidation or hydrolytic degradation of the fuels.

Procedure

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The test fuels as received varied widely in color, and one contained a precipitate which had settled to the bottom of the container. These solids were removed by filtration before further processing. A 100-ml specimen of each fuel was passed through a short column of freshly activated 100-200 mesh silica gel. In

all cases, the fuels emerging from the silica gel columns were colorless. The columns were then carefully rinsed with hexane to remove residual fuel. Isolated material was removed from the gel by sequential rinsing with chloroform and then methanol. The two eluants were combined and the solvents were removed by evaporation in a stream of prepurified nitrogen. The recovered materials varied from a colorless oily film to a dark yellow liquid.

Infrared absorption spectra were recorded for each of the isolates from the four test fuels and the control fuel.

Results

Infrared spectra for the isolates from fuels #1 and #2, shown in Figures 220 and 221, are typical of those obtained from polysulfideformal elastomers such as the Thiokols. A reference spectrum (ref. 12) of Thiokol LP62 is shown in Figure 222. Low molecular weight elastomer species are apparently extracted by the fuel.

The infrared absorption spectrum of the material isolated from fuel #5, shown in Figure 223, closely matches the spectrum of dibutoxyethyl adipate, Figure 224 (ref. 13), and appears to be that compound or a very similar one. This material is commonly used as a plasticizer.

The silica gel isolate from fuel #4, shown in Figure 225, can be characterized as a polyethylene oxide adduct of an alcohol or phenol derivative. A typical reference spectrum (ref. 14) for a material of this type, di-t-butyl phenol-EO_{10_12} adduct, is presented in Figure 226. Fuel #4 also contained a precipitate, the spectrum of which is shown in Figure 227. The spectrum is characteristic of a carboxylic acid salt and the material is probably a metal stearate.

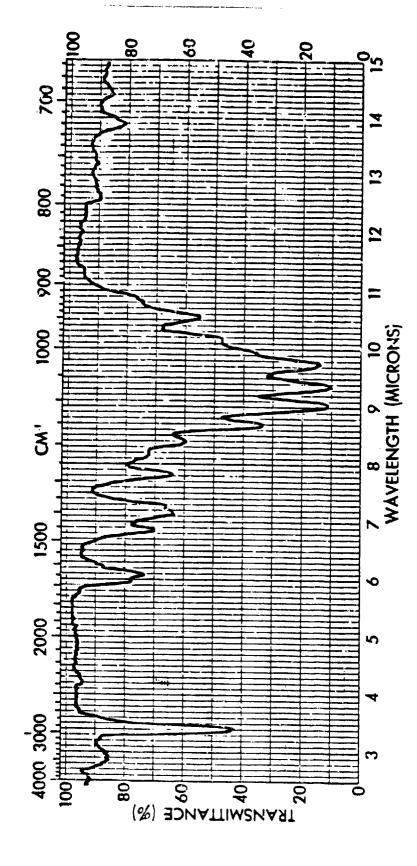


Figure 220. Infrared absorption spectrum of isolated material from JP-9 sample #1.

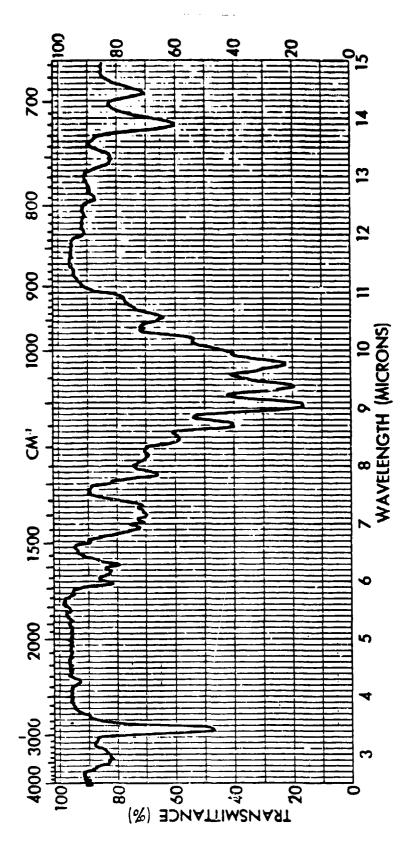


Figure 221. Infrared absorption spectrum of isolated material from JP-9 sample #2.

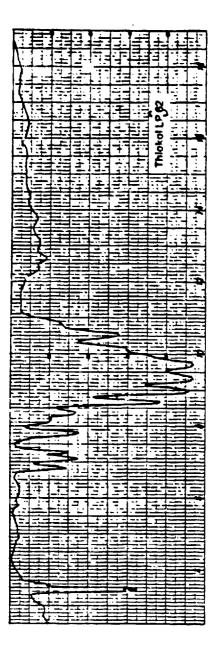


Figure 222. Infrared absorption spectrum of Thiokol LP62 polysulfide elastomer (ref. 12).

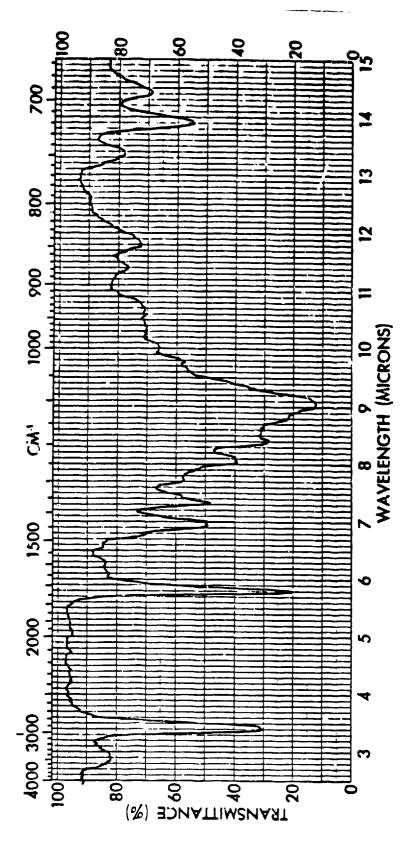


Figure 223. Infrared absorption spectrum of isolated material from JP-9 sample #3.

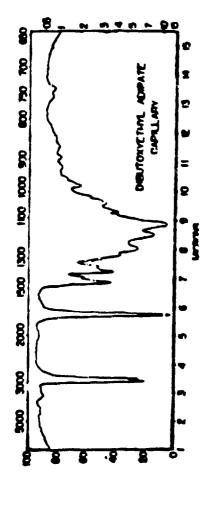


Figure 224. Infrared absorption spectrum of dibutoxyethyl adipate (ref. 13).

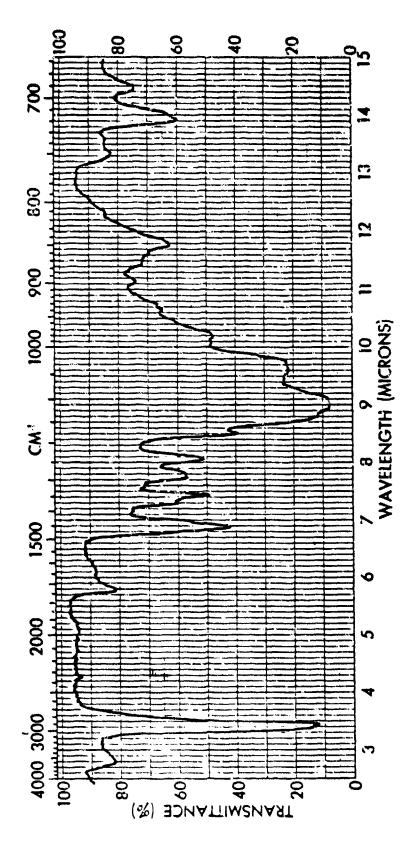
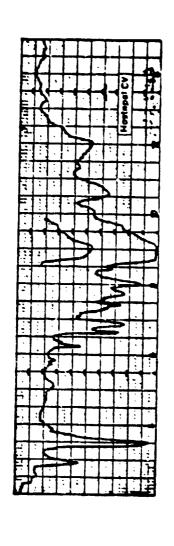


Figure 225. Infrared absorption spectrum of isolated material from JP-9 sample #4.



Infrared absorption spectrum of the ethylene oxide adduct of di-t-butyl phenol (ref. 14). Figure 226.

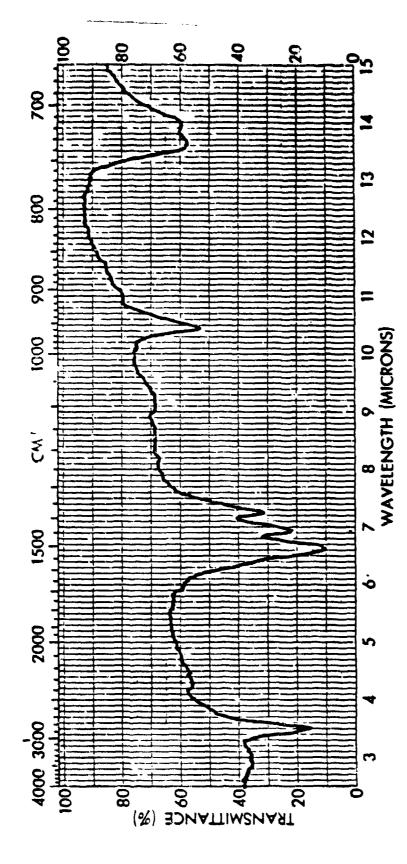


Figure 227. Infrared absorption spectrum of the pre-

The control fuel gave very little residue, and its spectrum shows the presence of some residual fuel and has some weak bands characteristic of JP-10 oxidation products as discussed in a previous report (ref. 15).

Data for the weights and descriptions of the isolated residues are given in Table 186 along with the identification of the major constituent of each residue.

Conclusions

The major constituents of the materials isolated from the fuels were characterized and all might logically be a part of an elastomer composition. Minor components could have been present and one cannot conclusively rule out the presence of small amounts of the fuel. However, it is most unlikely that the compounds listed in Table 186 could have resulted from any reaction or degradation of the fuels themselves.

TABLE 186. MATERIALS ISOLATED FROM ONE-HUNDRED MILLILITER PORTIONS OF JP-9 FUEL SAMPLES

Fuel	Weight of isolate, grams	Color of isolate	Identity
Control	0.0251	Colorless	Fuel and its degradation products
#1	0.2540	Yellow liquid	Low molecular weight polysulfide-formal elastomer species
#2	0.1426	Colorless oil	Low molecular weight polysulfide-formal elastomer species
#3	0.1577	Dark yellow liquid	Dibutoxyethyl adipate or similar compound
#4	0.2701	Dark yellow	Ethylene oxide adduct of alcohol or phenol derivative
#4	_a	Yellow precipitate	Carboxylic acid salt, likely a metal stearate

^aPrecipitate taken from sample container, not associated with the 100-ml portion examined.

2. PROPERTIES OF FUELS AND FUEL BLENDS

A multitude of high density fuels and fuel blends were analyzed for specific physical and chemical properties by procedures described in the Appendix. Density and specific gravity results are shown in Table 187. Kinematic viscosity results are shown in Table 188, and viscosity/temperature relationships are shown in Figures 228 through 233. Heat of combustion values are shown in Table 189, vapor pressure in Table 190, surface tension in Table 191, flash point in Table 192, autoignition temperature in Table 193, and fuel composition data in Table 194. Gas chromatograms from the fuel composition studies are presented in Figures 234 through 244.

TABLE 187. DENSITY AND SPECIFIC GRAVITY OF HIGH DENSITY FUELS

Sample description	Test temperature,	Density,	Specific gravity
PJ 5 Blends 50/50 Exo-Exo/Endo-Endo #1 50/50 Exo-Exo/Endo-Endo #2 50/50 Exo-Exo/Endo-Endo #3 RJ-5F	60 60 60 60		1.0712 1.0717 1.0714 1.0799
RJ-6 Blends AR 78024 AR 78024 AR 78024 Sample 1219 Sample 1220	-65 0 70 60 60	1.0754 1.0469 1.0169 1.0203 1.0223	
JP-9 Blends #1 (10% MCH*, 70% JP-10, 20% RJ-5 #2 (15% MCH, 55% JP-10, 30% RJ-5 Contaminated in shipment and filtered Contaminated in shipment and filtered Contaminated in shipment and filtered	70 70 32 70 100	0.9385 0.9421 1.0300 1.0133 0.9999	

^{*}Methylcyclohexane.

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TABLE 188. KINEMATIC VISCOSITY OF HIGH DENSITY FUELS

Sample description	-65°F	Kinematic viscosity, centistokes -40°F 77°1	viscosity 0°F	, centis	tokes 77°F	210°F
NJ-5 Fuel Blends 50/50 Exo/Endo #1 50/50 Exo/Endo #2 50/50 Exo/Endo #3	9162 9173 9056	1231 1242 1219		;	15.97 16.03 15.94	
(J-5F			189.5	22.60		3.105
AJ-6 Blends AR 78024 Sample 1219 Sample 1220	437.1 438.0 459.0		37.92	8.80		-
JP-9 Blends #1 (10% MCH*, 70% JP-10, 20% RJ-5) #2 (15% MCH, 55% JP-10, 30% RJ-5) Contaminated in ship.ent and filtered	-30 43.59 49.33 893.3	17.34 18.81 150.2	51.53			

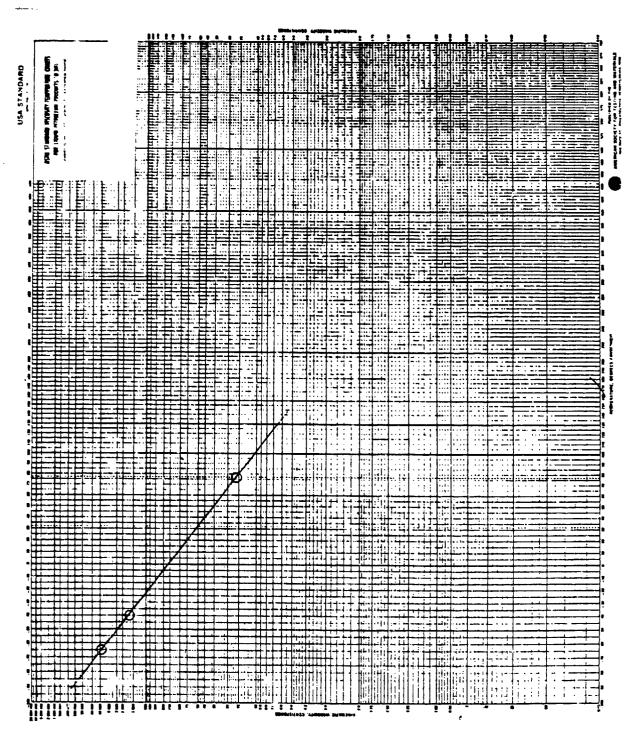


Figure 228. Viscosity/temperature plot for sample #1, 50% Exo-Exo, 50% Endo-Endo.

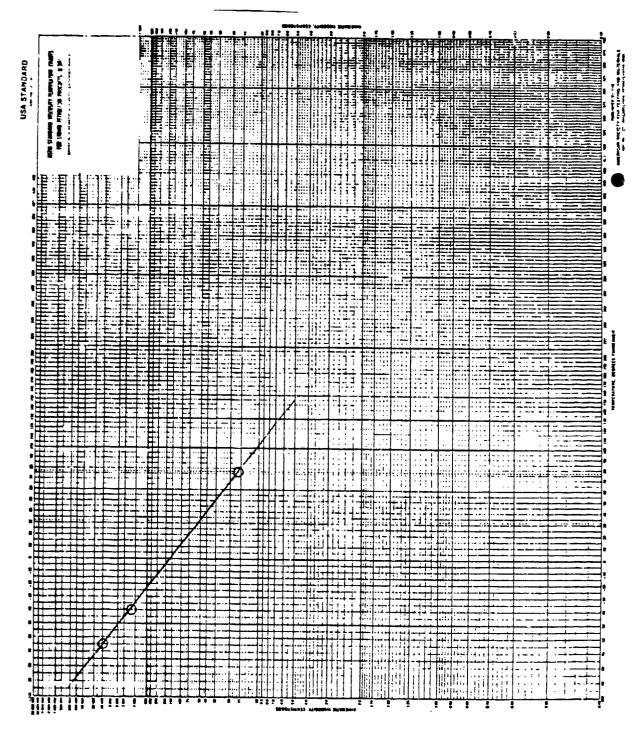


Figure 229. Viscosity/temperature plot for sample #2, 50% Exo-Exo, 50% Endo-Endo.

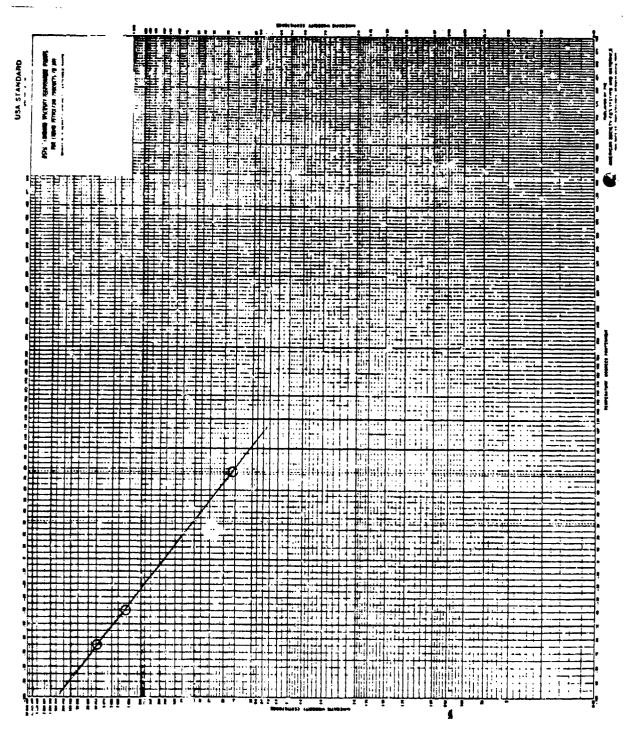
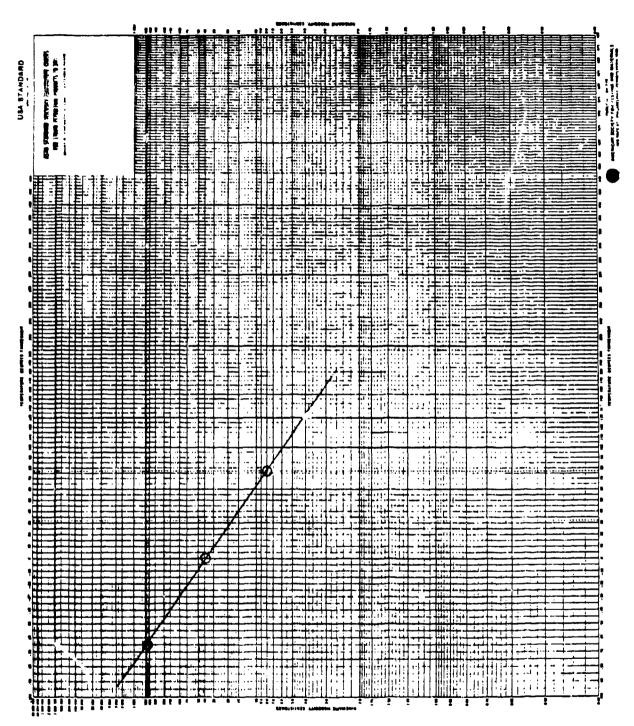


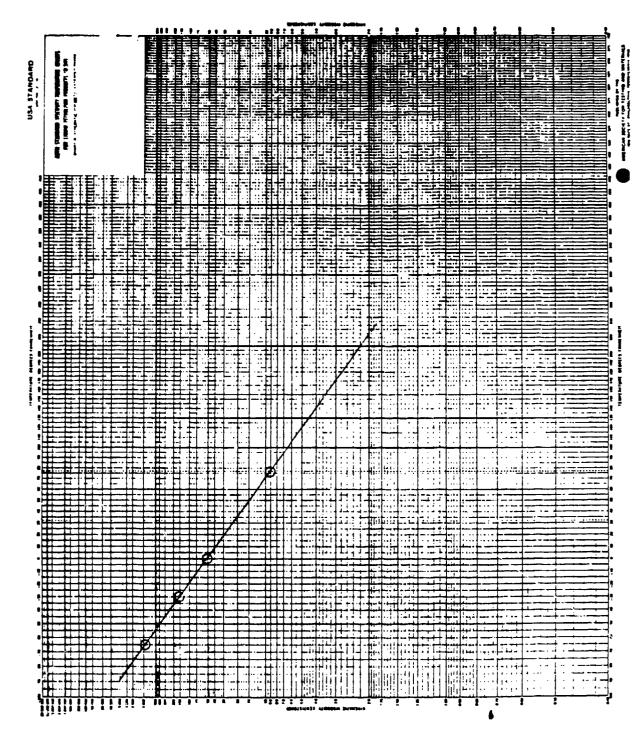
Figure 230. Viscosity/temperature plot for sample #3, 50% Exo-Ex0, 50% Endo-Endo.

Figure 231. Viscosity/temperature plot for RJ-5F.



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Figure 232. Viscosity/temperature plot for RJ-6.



. . .

Figure 233. Viscosity/temperature plot for JP-9 (contaminated and filtered).

TABLE 189. HEAT OF COMBUSTION OF HIGH DENSITY FUELS

	Gross values,	Btu/lb	Net average,
Sample identification	Duplicates	Average	Btu/lb
RJ-5 Fuel Blends			
50/50 Exo/Endo #1	18,751; 18,745	18,748	17,845
50/50 Exo/Endo #2	18,748; 18,755	18,752	17,849
50/50 Exo/Endo #3	18,764; 18,748	18,756	17,853
RJ-5P	18,686; 18,679	18,683	17,780
RJ-6 Fuel Blends			
Sample 1219	18,913; 18,954	18,934	17,923
Sample 1220	18,886; 18,899	18,893	17,882

TABLE 190. VAPOR PRESSURE OF HIGH DENSITY FUELS

Sample identification	Temperature, °F	Pressure, torr
RJ-6 Fuel Blends		
Sample 1219	300	147.0
Sample 1220	300	138.0
JP-9 Fuels Blends		
#1 (10% MCH ^a , 70% JP-10, 20% RJ-5) #2 (15% MC 55% JP-10, 30% RJ-5)	70 70	12.0 16.5

aMethylcyclohexane.

TABLE 191. SURFACE TENSION OF HIGH DENSITY FUEL

		iace tens dynes/cm	sion
<u>le</u>	-65°F	0°F	70°F
RJ-6 AR78024	39.04	36.15	34.07

TABLE 192. FLASH POINT OF HIGH DENSITY FUELS

	Temp	erature
Sample description	°F	°C
RJ-5F	216	102.5
JP-9 Fuel Blends		
#1 (10% MCH, a 70% JP-10, 20% RJ-5)	86	29.8
#2 (15% MCH, 55% JP-10, 30% RJ-5)	66	18.8

aMethylcyclohexane.

TABLE 193. AUTOIGNITION TEMPERATURE OF HIGH DENSITY FUELS

		Autoig temper	
Fuel	Components	°C±2.5	°F±4.5
JP-9	69.97% JP-10 20.02% RJ-5G 10.00% MCH	250.1	482
JP-10	Exo-THD	245.7	474
a _{Methy}	lcyclohexane.		

TABLE 194. GAS CHROMATOGRAPHIC ANALYSIS OF TEST FUEL BLENDS

	Retention Time,	Area Percents of Components a								
Sample	minutes	Blend 1	Blend 2	Blend 3						
Three RJ-5 fuel blends made	4.14	0.56	0.31	0.45						
up of 50% Exo-Exo plus 50%	6.07	0.50	0.37	0.32						
Endo-Endo	7.55	0.60	-	-						
	8.54	1.17	1.14	0.93						
	11.50	0.31	•	-						
	12.95	0.37	0.62	0.32						
	16.06	2,22	3.21	3.88						
	16.25	0,71	0.80	0.97						
	16.51	43.61	43.33	43.59						
	16.96	0.93	0.87	0.91						
	17.31	47.54	46.95	46.98						
	17.65	0.50	1.31	0.62						
	18.75	0.66	0.74	0.42						
	19.52	0.56	0.37	0.57						
RJ-5, 22 December 1978	15.51	1,260								
	15.68	8.617								
	16.00	19,208								
	16.43	0.676								
	16.91	70.235								
RJ~5F	17.24	3,548								
	17.48	25.265								
	17.51	8,234								
	17.71	13,199								
	17.77	0.932								
	17.87	1.180								
	18.15	2,567								
	18.57	44,513								
	18.90	0.561								
		Blend 1219	Blend 1220							
Two RJ-6 Fuel blends	13.45	45,111	40.577							
140 10-0 1 461 1/20142	14.14	1.171	1.210							
	Total JP-10	46.282	41.787							
	21.79	0.858	1.113							
	21.91	4.291	4.452							
	22.20	8.793	9.459							
	22.65	0.303	0.331							
	23.08	39.273	42.618							
	24.20	0.096	0.110							
	24.85	0.113	0.126							
	Total RJ-5	53,727	58.209							
	TOCAT WAS	JJ, 12,	301203							

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TABLE 194 (continued)

0	Retention Time,	Area Percents	of Componentsa			
Sample	minutes	Batch 3,8383-12	Batch 4,8353-13			
Two RJ-6 fuel blends	13.12	37.90	38.27			
	13.74	0.93	0.97			
	Total JP-10	38.83	39.24			
	21.24	1.57	1.59			
	21.39	5.58	5.68			
	21.69	9.06	9.06			
	22.17	0.41	0.00			
	22.59	44.53	44.44			
	Total RJ-5	61.15	60.77			
Company and a 2D O	4.24 MCH ^b	6 72				
Contaminated JP-9 after filtering	13.92	6.72 1.71				
arter firefrud	14.30	0.64				
	14.16	0.28				
	14.58	0.70				
	14.73	0.47				
	14.88	1.67				
	14.96	0,22				
	15.06	4.77				
	15.23	4.14				
	15.39	0.63				
	16.21	6.47				
	Total RJ-4	21.70				
	20.01	0.49				
	20.15	7.60				
	20.45	12.70				
	20.94	0.79				
	21.33	50.00				
	Total RJ-5	71.58				
Two JP-10 Fuels		Not distilled	Distilled			
Sample distilled for DOE	11.46	98.300	99,233			
	11.76	9,266	0.151			
~	12.02	1.118	0.615			
	12.18	0.316				
Sample distilled for a standard	8.36		99.069			
	8.59		0.133			
	8.78		0.659			
	8.90		0.139			

a Chromatographic peak area percents are essentially the same as weight percents, since all isomers should have the same response with a flame ionization detector. b Methylcyclohexane.

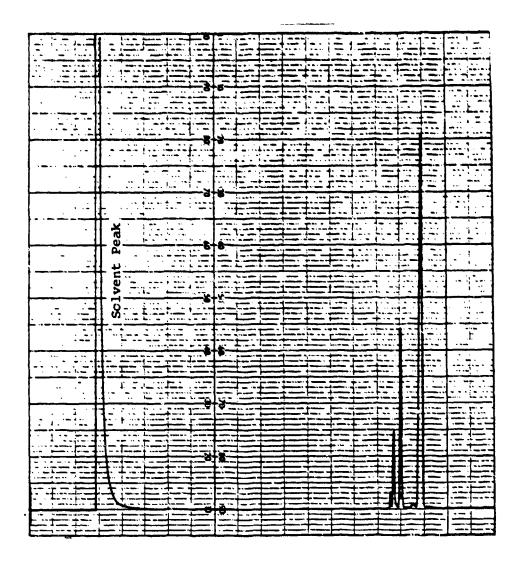


Figure 234. Gas chromatogram of RJ-5 (12-22-78).

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Figure 235. Gas chromatogram for 50% Exo-Exo, 50% Endo-Endo #1, RJ-5.

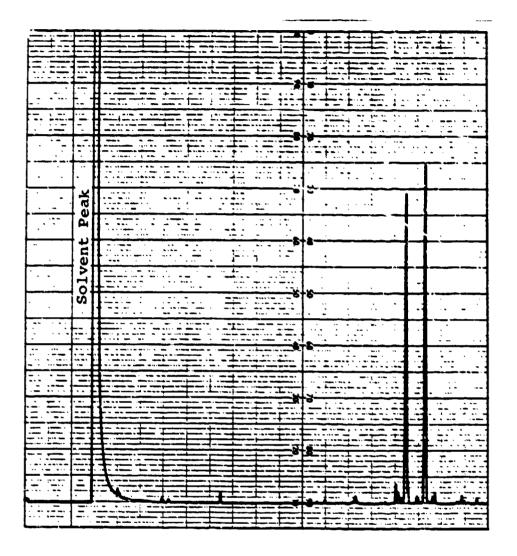


Figure 235. Gas chromatogram for 50% Exo-Exo, 50% Endo-Endo #2, RJ-5.

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Figure 237. Gas chromatcgram for 50% Exo-Exo, 50% Endo-Endo #3, RJ-5.

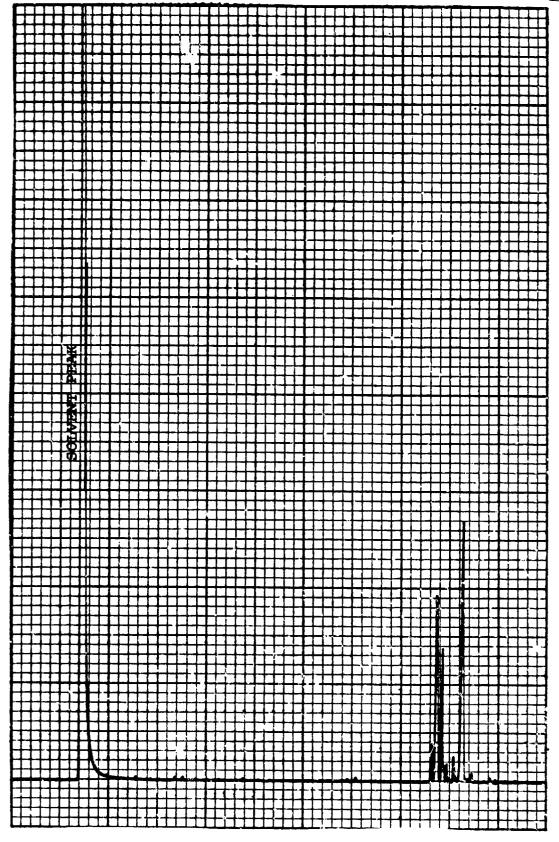


Figure 238. Gas chromatogram of RJ-5F.

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Figure 239. Gas chromatogram of RJ-6 Blend #1219.

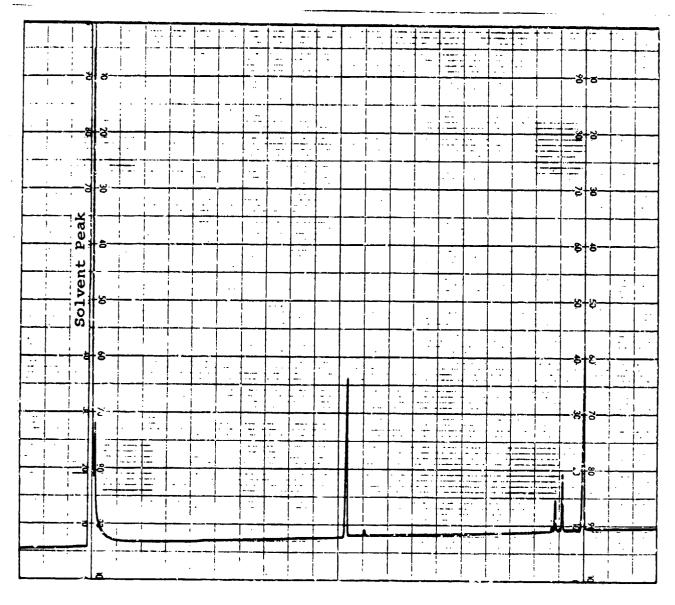


Figure 240. Gas chromatogram of RJ-6 %lend #1220.

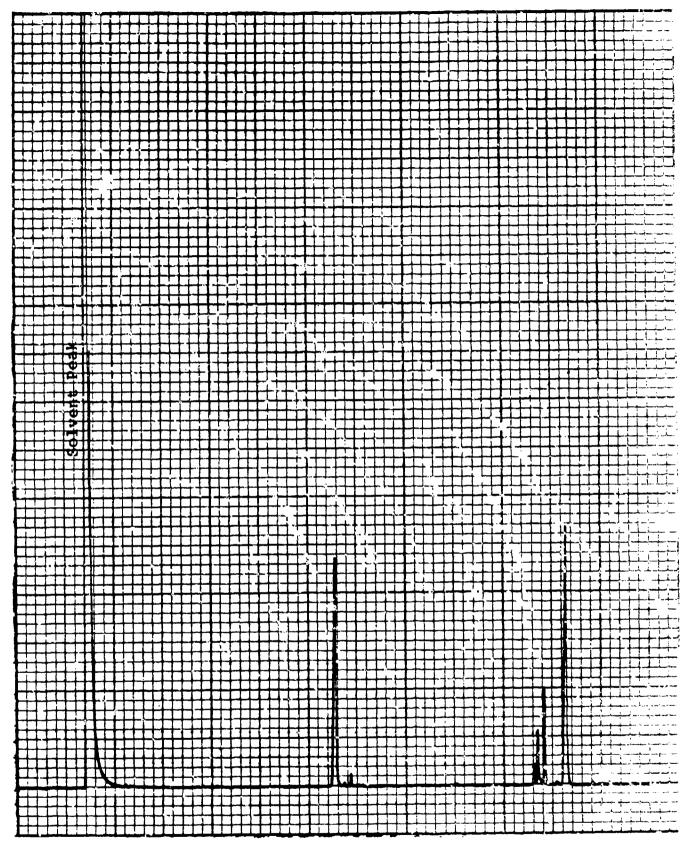


Figure 241. Gas chromatogram of RJ-6 fuel, Batch 3, 8353-12.

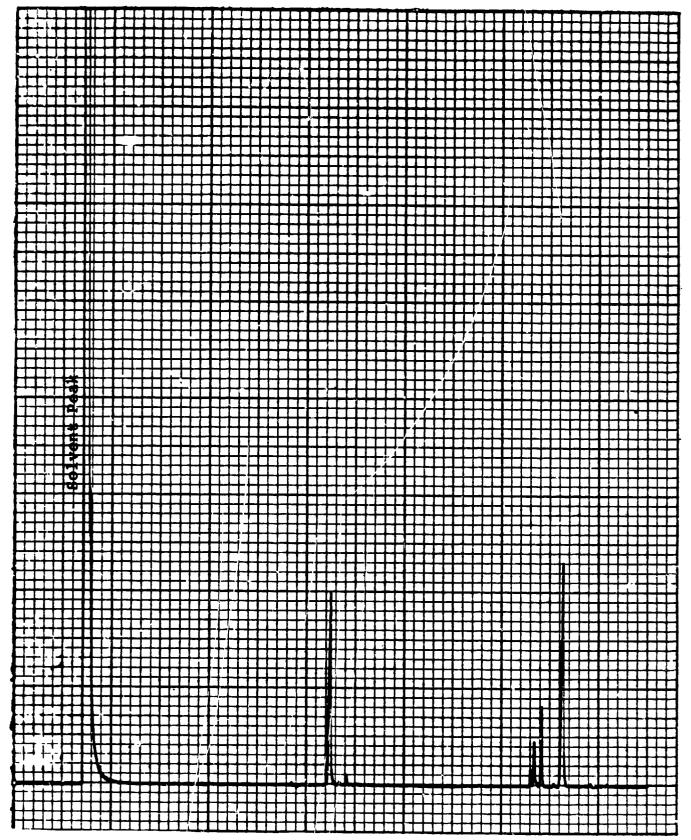


Figure 242. Gas chromatogram of RJ-6 fuel, Batch 4, 8353-13.

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Figure 243. Gas chromatogram of JP-10 after distillation for standard preparation.

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Figure 244. Gas chromatogram of JP-10 after distillation for DOE.

3. PHYSICAL PROPERTIES OF JP-10

Various physical properties of JP-10 as a function of temperature were required by ALCM design engineers. Density, surface tension, thermal conductivity, and air and water solubility were determined at temperatures between -66°F and 160°F using the analytical procedures described in the Appendix. The results are shown in Table 195.

TABLE 195. VARIOUS PROPERTIES OF JP-10

Temperature °F	Density g/cc	Surface tension dyne, cm ⁻¹	Thermal conductivity Btu/ft/hr/°F	Air solubility Ostwald solubility coefficients	Water solubility ppm, vol
-65	0.9926	37.850	0.07168	-	-
-60	-	_	-	0.098	-
-40	-	_	0.07055	-	-
-30	0.9772	35,928	~	_	-
14	-	-	0.06796	-	-
32	0.9500	32.926	_	_	26
68	_	-	0.06551	-	-
72	-	30.534	-	0.129	-
74	-	-	•	~	29
77	0.9303	-	**	-	-
110	•	-	-	_	47
140	_	-	-	0.140	a-ra
151	~	-	0.06128	-	-
155	0.8960	-	-	-	_
158	•	26.097	-	-	-

Note: Dash indicates no measurement made.

4. PHYSICAL PROPERTIES OF RJ-6

Various physical properties were determined for a sample of RJ-6 fuel in support of ASALM programs. All test methodology is described in the Appendix. Surface tension, density, vapor pressure, kinematic viscosity, specific heat, thermal conductivity, dielectric constant, and water solubility were determined at -20, 32, 70, and 100°F; and these data are shown in Table 196. Air

aml of air/ml of fuel, both volumes measured at the temperature of saturation.

solubility was determined at -60, -20, 70, and 140°F by mass spectrometry, and these results are shown in Table 197. Heat of combustion, flash point, and autoignition temperature data are shown in Table 198. The results of a composition analysis by gas chromatography are shown in Table 199.

TABLE 196. PHYSICAL PROPERTIES OF RJ-6 AT FOUR TEMPERATURES

	-20°F	32°F	70°F	100°F
Surface tension, dynes/cm	38.59	35.50	33.27	31.74
Density, g/cc	1.0499	1.0269	1.0106	0.9973
Vapor pressure, mm Hg	. -	5.0	9.0	13.0
Kinematic viscosity, centistokes	61.30	16.110	8.160	5.315
Specific heat, cal/g/°K	0.285	0.344	0.398	0.443
Theimal conductivity, Btu/ft/hr/°F	0.0662	0.0649	0.0638	0.0630
Dielectric constant	2.395	2.354	2.319	2.297
<pre>Water solubility, ppm (vol)</pre>	-	31	52	65

TABLE 197. AIR SOLUBILITY IN RJ-6 FUEL SPECIMEN

Tempe	rature, T	Ostwald solubility
°F	(°C)	Ostwald solubility coefficient
-60	(-51)	0.051
-20	(-29)	0.079
70	(21)	0.089
140	(60)	0.096

aml of air/ml of fuel, both at temperature, T.

TABLE 198. COMBUSTION RELATED PROPERTIES OF RJ-6

Heat of combustion, ASTM D 240-64

Gross, Btu/lb Net, Btu/lb	18,892 17,903		
Flash point, ASTM D 93-71	62°C (144°F)		
Autoignition temperature ASTM D 2155-66	241.2°C (466.2°F)		

TABLE 199. GAS CHROMATOGRAPHIC ANALYSIS OF RJ-6

Retention time, min	Weight, %			
1.4.15	42.1 (Total JP-10)		
22.07 22.23 22.50 22.98 23.31	3.4 17.2 12.5 3.3 21.5	RJ-5 Isomers (Total = 57.9%)		

5. GC/MS ANALYSES OF JP-9/AIR SAMPLES FROM A FLAMMABILITY TEST CELL

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Two vapor samples contained in glass gas bottles and taken from a test cell used to determine flammability of fuel vapors were analyzed for hydrocarbon concentration. The hydrocarbons suspected to be present were the three components of JP-9 fuel. The vapor samples were analyzed by GC/MS against a standard of JP-9 prepared in methanol. The results obtained in micrograms were converted to percent concentrations using the ideal gas law equation.

Analytical Procedure

Prior to analysis the gas bottles were warmed to ensure that all vapor components were in the gas phase. The two vapor samples were then analyzed on an HP-5982 GC/MS using the following GC conditions.

Column: 6 ft x 1/8 in. glass packed with Tenax

Column Temperature: 2 minute at -30°C, then program to

280°C at 16°C/minute

Carrier Flow: helium at 30 ml/minute

Sample Size: 10 ml of vapor at 25°C and 760 mm

Analytical Results

The only hydrocarbons detected in the gas bottles were the components of JP-9 fuel. The weights of the components detected in ten-milliliter volumes of each gas sample are shown in Table 200.

TABLE 200. GC/MS ANALYSIS OF JP-9 COMPONENTS IN TWO GAS SAMPLES

	Concenti 10-ml gas	cation in
	Bottle 1,	Bottle 2,
JP-9 component	μg	<u>ha</u>
Methyl cyclohexane (MCH)	300	12
Tetrahydrodicyclo pentadiene (JP-10)	100	2
Hydrogenated norbornadiene dimers (RJ-5)	50	20

The total weight of JP-9 fuel detected in the 10-ml gas specimen could be converted to volume percent using the ideal gas law:

$$PV = \frac{qRT}{m}$$

where P = pressure in atmospheres

V = volume in liters

g = weight in grams

m = molecular weight

 $R = 0.08202 \text{ 1-atm deg}^{-1} \text{ mole}^{-1}$

T = temperature in degrees Kelvin = 302°K

The volume occupied by the JP-9 vapors could be determined by solving for V. Using this volume and the original volume of the sample analyzed (10 ml), volume percents of JP-9 vapors in the gas samples were calculated. The average molecular weight of the JP-9 vapors in each gas sample was calculated from the component molecular weights and their percentages of the total fuel. All results are shown in Table 201.

TABLE 201. DETERMINATION OF VOLUME PERCENT JP-9 IN GAS SAMPLES

Gas sample	Wt. of JP-9 in 10-ml sample, µg	Calculation average MW	Volume of JP-9, ml	Volume %
Bottle 1	450	116	0.096	1.0
Bottle 2	34	115	0.005	

6. GC ANALYSES AND PHYSICAL PROPERTY MEASUREMENTS FOR THREE ASHLAND RJ-5 FUELS

Three RJ-5 fuels were analyzed for selected physical and chemical properties. The fuels, which were labeled 1, 2, and 3, were all from Ashland Lot B-9-78. The specific analyses are described below:

<u>Specific gravity</u> was determined by the dilatometer method in which the volume of a known weight of fuel was measured at 60°F.

Results are shown in Table 202.

TABLE 202. SPECIFIC GRAVITY, 60/60°F

Fuel	Values at 60°F
B-9-78-1	1.0836
B-9-78-2	1.0833
B-9-78-3	1.0836

Kinematic viscosity was determined at four temperatures by ASTM Method D 445. The test results are presented in Table 203 and graphs illustrating viscosity as a function of temperature are presented in Figures 245 through 247.

TABLE 203. KINEMATIC VISCOSITY

	Centistokes						
Fuel	-20°F	32°F	70°F	100°F			
B-9-78-1	522.6	65.89	24.77	13.66			
B-9-78-2	526.1	65.95	24.79	13.66			
B-9-78-3	523.2	65.92	24.77	13.66			

Gross and net heat of combustion were determined by oxygen bomb calorimetry using ASTM Method D 240 and hydrogen content data from AFWAL/POSF. The test values are shown in Table 204.

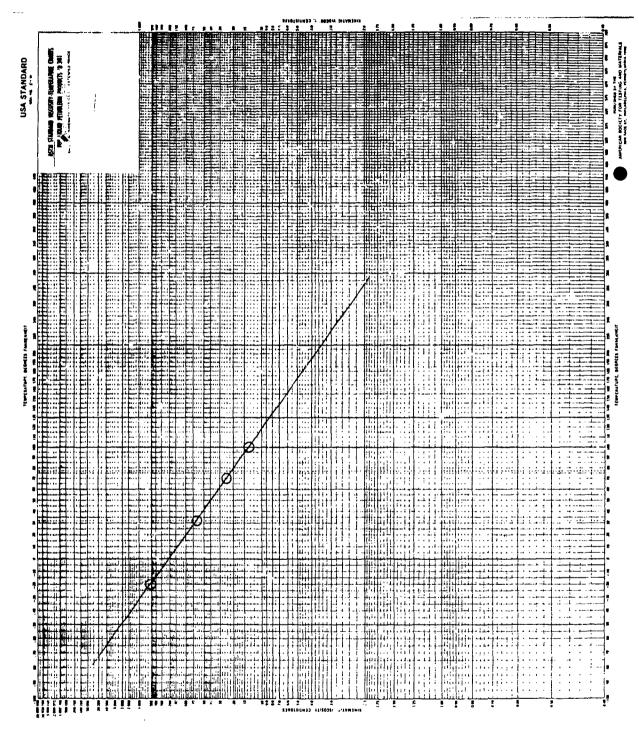
TABLE 204. HEAT OF COMBUSTION

	G	ross, Btu	Net, Btu/lb	3	
Fuel	Duplio	cates	Average	Average	% н ^а
B-9-78-1	18,834	18,820	18,827	17,919	9.95
B-9-78-2	18,812	18,846	18,829	17,921	9.95
P-9-78-3	18,815	18,806	18,811	17,903	9.95

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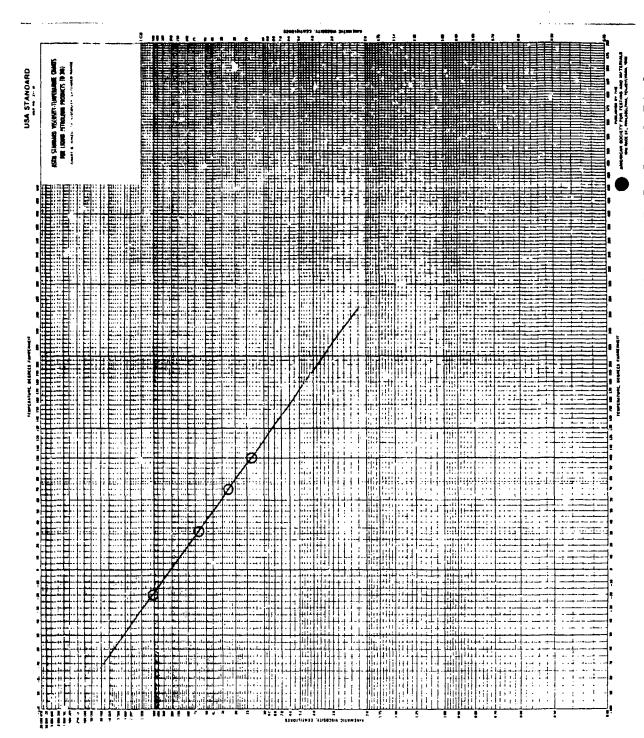
Gas chromatographic analyses were conducted to determine purity and isomer ratios for the fuels. A Perkin-Elmer Model 3920B gas chromatograph fitted with a 50-meter glass capillary column coated with SF-96 stationary phase was employed for the analyses. The analytical data shown in Table 205 were obtained from a Hewlett-Packard 3350 laboratory data system. The gas chromatograms for the three fuels are reproduced in Figures 248 through 250.

apercent hydrogen data from AFWAL/POSF used to calculate net heat of combustion.

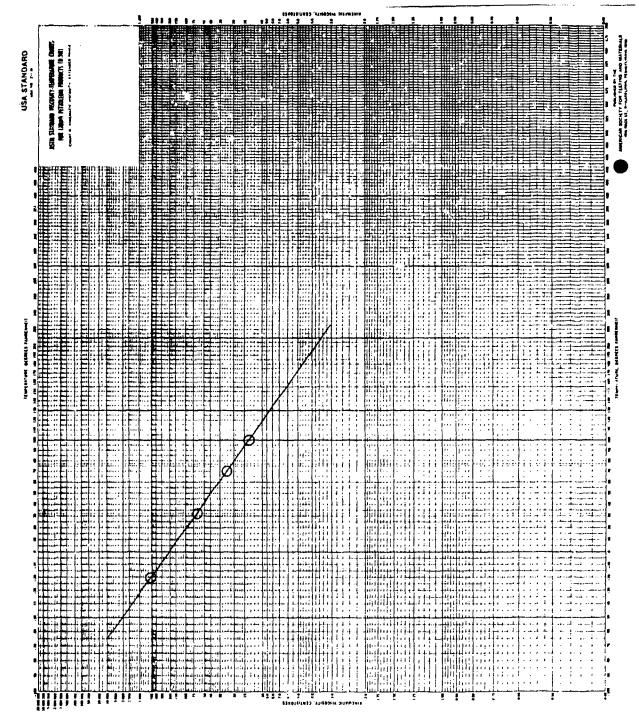


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Viscosity/temperature plot for JP-5 fuel B-9-78-1



Viscosity/temperature plot for RJ-5 fuel B-9-78-2 Figure 246.



Viscosity/temperature plot for RJ-5 fuel B-9-78-3

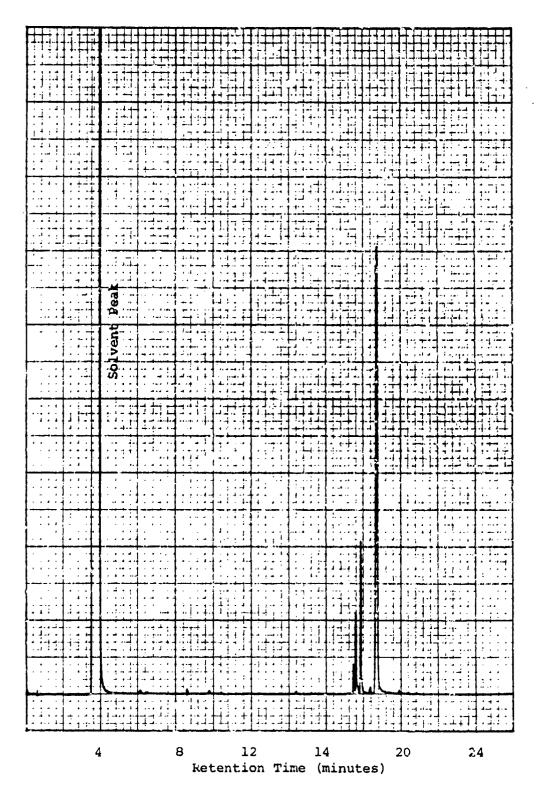
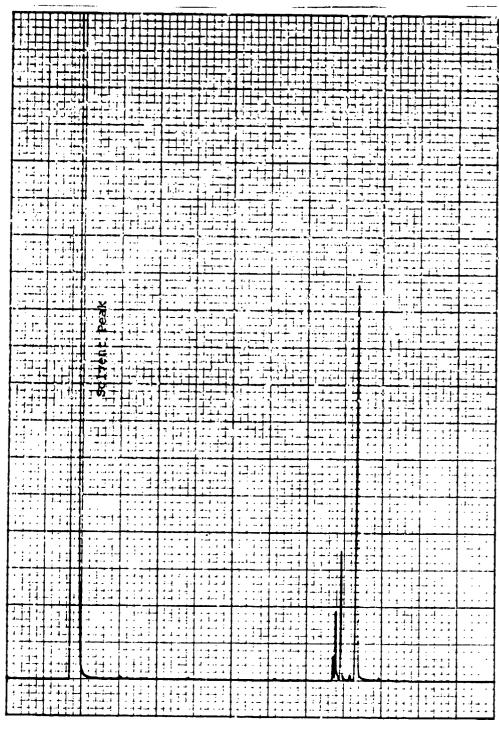


Figure 248. Gas chromatogram of RJ-5 Ashland fuel B-9-78-1.



4 8 12 14 20 24 Retention Time (minutes)

Figure 249. Gas chromatogram of RJ-5 Ashland fuel B-9-78-2.

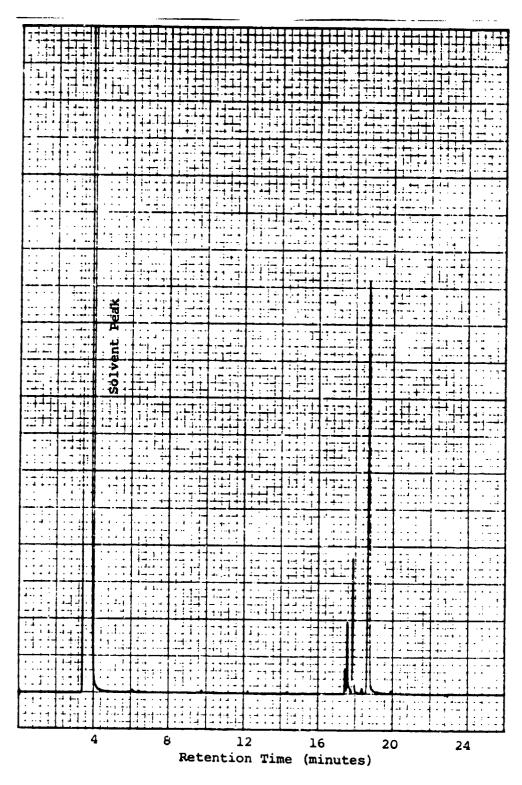


Figure 250. Gas chromatogram of RJ-5 Ashland fuel B-9-78-3.

TABLE 205. GAS CHROMATOGRAPHIC ANALYSES

Retention time	Area parcents of components in fuels			
minutes	B-9-78-1	B-9-78-2	B-9-78-3	
6.10 17.49 17.61 17.90 13.39 18.77 19.95	0.40 2.64 9.54 15.78 0.68 70.70 C.26	0.33 2.62 9.48 15.54 0.68 71.10 0.25	0.32 2.58 9.43 15.31 0.69 71.42 0.25	

^aEssentially the same as weight percents for these fuels.

7. VISCOSITY AND SURFACE TENSION OF RJ-4, JP-9, AND JP-10 FUELS WITH AND WITHOUT FERROCENE ADDITIVE

Kinematic viscosity and surface tension were measured at 77°F and 100°F for JP-9, JP-10, and RJ-4 jet fuels. The same measurements were then made on these fuels after addition of ferrocene at the 1,300 ppm level (400 ppm iron). The purpose of the investigation was to determine if this level of ferrocene had any effect on the physical properties. The test results are shown in Tables 206 and 207 below.

TABLE 206. KINEMATIC VISCOSITY OF FUELS WITH AND WITHOUT FERROCENE

	Centistokes					
Test fuel:	JP-9, B	atch 38	JP-10,	Drum 1	RJ	-4
Test temperature, °F:	77	100	77	100	77	100
Neat fuel: Ferrocene added:	3.154 3.157	2.488 2.488	2.995 2.996	2.380 2.379	4.857 4.857	3.620 3.618

TABLE 207. SURFACE TENSION OF FUELS WITH AND WITHOUT FERROCENE

	Dynes per centimeter					
Test fuel:	JP-9, Batch 38	3 JP-10, Drum 1	RJ-4			
Test temperature, °F:	77 100	77 100	77 100			
Neat fuel:	30.89 30.04	31.86 30.31	30.90 29.53			
Ferrocene added:	31.09 29.91	32.22 30.44	31.04 29.79			

The data show that the addition of ferrocene to these fuels did not have a significant effect on the physical properties in question. Examination of the surface tension data shows a slight change in surface tension values for fuels with the additive. However, the magnitude of the changes are far less than is found between various lots of RJ-5 or between various compositions within the JP-9 specification range (ref. 15, Table 8, page 14).

8. PREPARATION OF JP-10 AND RJ-5 FUEL STANDARDS BY FRACTIONATION

Highly pure JP-10 and RJ-5 fuel samples were required for use as standards in accordance with MIL-P-87107B specification. Consequently, JP-10 fuel from Drum 31 and RJ-5 fuel from Patch #3 were fractionated on a 42-inch Todd Vigreux column under vacuum. A pressure stabilizing apparatus was used to maintain the selected vacuum. After the distillations the purified fuels were placed into GC septum vials and then analyzed for purity on a capillary gas chromatographic system prior to sample distribution. Included in the following report sections are the fractionation parameters for each fuel, the GC analysis conditions and results for the determination of percent purity in each fuel, representative chromatograms, and the sample bottling procedure and distribution locations.

Distillation of JP-10

About 250 milliliters of JP-10 was distilled at a 10:1 reflux ratio under a vacuum of about 30 mm. This vacuum was selected to keep the boiling temperature below 100°C and avoid possible cracking of the hydrogenated cyclopentadiene dimer. Successive 5-milliliter fractions were collected until the refractive indices of at least three adjacent cuts agreed within ±0.0002, and then a 50 milliliter fraction was collected to be used as the standard. The boiling ranges and refractive indices of the collected fractions are shown in Table 208.

TABLE 208. DISTILLATION OF JP-10

Fraction number	Fraction size, ml	Boiling range, °C/millimeters	Refractive index, N _D ²⁵	Disposition of sample
1 2	5 5	83-84/29 8 4- 85.5/29	1.4821 1.4848	Discarded Discarded
3	5	85/29	1.4863	Discarded
4 5	5 5	86/29 85.5/29	1.4868 1.4867	Discarded Discarded
6	5	85/29	1.4869	Discarded
7	5	85/29	1.4868	Discarded
8	5	85/29	1.4869	Discarded
ò	50	86.5-87/30-31	1.4869	Kept as standard
Residuum	~160	-	-	Discarded, still clear color

The starting fuel had a refractive index of 1.4872, which compares favorably with the value for the 50 milliliter fraction kept as a standard.

It can be observed that some of the higher numbered fractions had lower indicated boiling temperatures than lower numbered fractions. This was due to variations in the vacuum system which were too small to be recorded.

Distillation of RJ-5 Fuel

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About 100 milliliters of RJ-5 was distilled at an 8:1 reflux ratio under a vacuum of about 50 milliliters. Almost the entire fuel was fractionated this time so as not to significantly change the isomer ratios present in the fuel. Successive 5-milliliter fractions were collected until only 6.9 grams of a dark yellow and viscous residuum remained. The boiling ranges and refractive indices of the collected fractions are shown in Table 209.

TABLE 209. DISTILLATION OF RJ-5

Fraction number	Fraction size, ml	Boiling range, °C at 50 mm Hg	Refractive index, N _D ²⁵	Disposition of sample
1	5	165-166.5	•	Heterogeneous mixture, cloudy; discarded
2	5	166.5-167.5	1.5360	Discarded
3	5	167.5-168	1.5370	Discarded
4	5	168-168.5	1.5370	Discarded
5	5	168.5-169	1.5381	Retained
6	5	169	1.5382	Retained
7	5	169	1.5391	Retained
8	5	169	1.5395	Retained
9	5	169	1.5395	Retained
10	5	169	15399	Retained
11	5	169-170	5407	Retained
12	5	170	5412	Retained
13	5	170-171	15419	Retained
14	5	171	1.5424	Retained
15	5	171	1.5424	Retained
16	5	171	1.5429	Retained
17	5	171-171.5	1.5433	Retained
18	5	171.5-172	1.5436	Retained
19	5	172	1.5438	Retained
20	5	172	1.5439	Retained
Residuum	6.9 grams	>210		Dark yellow and viscous, discarded

Fractions 5-20 were combined to make the purified standard. This mixture had a refractive index of 1.5414 as compared to 1.5412 for the starting fuel. The fact that the two refractive indices are so close indicates that the isomer ratios were not significantly changed.

GC Analysis of Purified Fuels

Gas chromatographic analyses were conducted on the purified fuels using a Perkin-Elmer Model 3920B instrument fitted with a 50-meter by 0.01-inch glass open tubular column coated with SF-96 stationary phase. Other parameters include:

Type detector - flame ionization

Carrier gas - 1 ml/min nitrogen

Column temperature - program 50-200°C at 8°C/min

Injection port - 200°C

Detector temperature - 200°C

Recorder - 1 mV

Electrometer range - 10

The data were recorded and processed using a Hewlett-Packard 3350 laboratory system. The calculated percent purity values for the distilled fuel were 98.8 for JP-10 and 99.2 for RJ-5. In each case minor impurities which have boiling points very close to the major fuel component(s) were not removed by distillation.

The gas chromatogram for the distilled JP-10 fuel is presented in Figure 251. The gas chromatogram for distilled RJ-5 is shown in Figure 252. The gas chromatogram for the undistilled RJ-5 (not shown) was similar in shape to Figure 252, which confirmed that the distillation process did not significantly alter the isomer ratios.

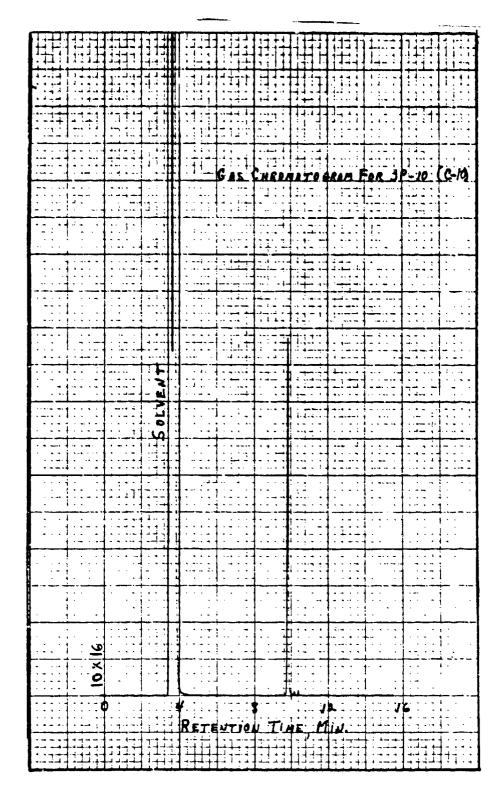
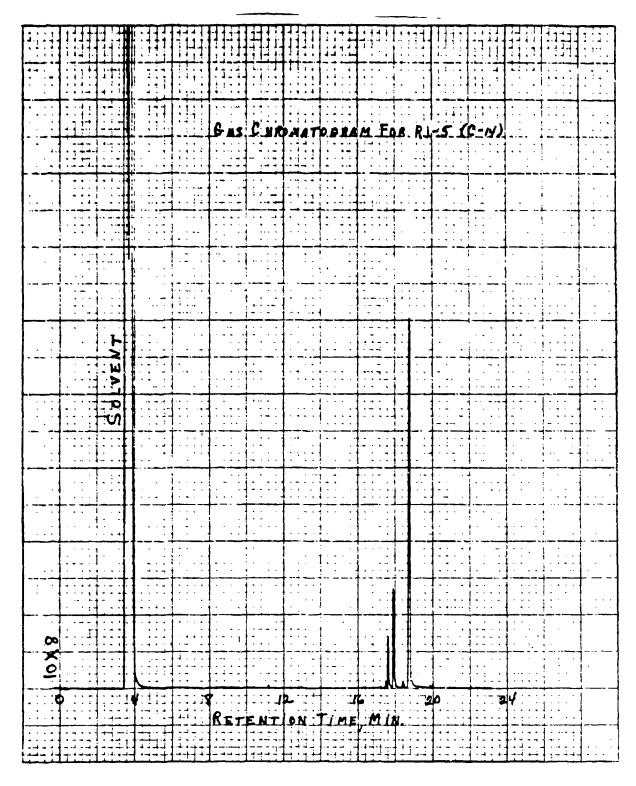


Figure 251. Gas chromatogram for distilled JP-10 fuel, labeled "C-10."



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Figure 252. Gas chromatogram for distilled RJ-5 fuel, labeled "C-14."

Sample Distribution

Ten 1-milliliter septum-capped vials were prepared with each distilled fuel. Each cap contained a Teflon-lined silicone septum. The caps were crimped over the lips of the glass vials to make a seal.

The ten JP-10 vials were each labeled "C-10." The ten RJ-5 vials were each labeled "C-14." One each of the vials was then sent to Tinker AFB, Oklahoma (Attn: P. K. Meredith), along with representative chromatograms. The other 18 vials of fuel and chromatograms were sent to Mr. J. McCoy at AFWAL.

9. PHOSPHORUS ANALYSES OF JP-9 FUEL SAMPLES BY GC

Samples of JP-9 fuel required to meet ALCM fuel delivery schedules were analyzed for phosphorus content using a gas chromatograph equipped with a phosphorus-specific detector. The analyses were conducted on a Varian Model 3700 gas chromatograph equipped with a Thermionic Specific Detector (TSD), which is 10^5 times more sensitive toward phosphorus-containing compounds than hydrocarbons. The analysis conditions are recorded below.

Analytical Column: 4 ft x 1/8 in. I.D. glass containing 5%

SE30 on 70/80 mesh Anakron ABS

Column Temperature: 210°C

Hydrogen: 5 ml/min at 20 psig

Air: 175 ml/min at 50 psig

Carrier Gas: 40 ml/min helium

Injection Port Temp.: 250°C

Detector Temp.: 250°C

Bias Voltage: 550 volts

Electrometer Sensitivity: Range 10⁻¹² x 256 attenuation

Recorder: 1 mV full scale with 0.5 cm/min

chart speed

Minimum Detection Limit: 0.3 ppm

The analyses were conducted with triphenylphosphine (11.8% P, 99+% pure) in JP-9 standards containing 40, 20, 10, 3.2 and 0.5 ppm phosphorus. The analytical results are shown in Table 210.

TABLE 210. PHOSPHORUS IN JP-9 FUEL SAMPLES

Submitted JP-9 samples	Phosphorus, ppm
32 ppm Phosphorus from TPPa 16 ppm Phosphorus from TPPa 8 ppm Phosphorus from TPPa 17-9 [Blank] 81-406M, 81-F-483 81-417m, 81-F-499 81-418m, 81-F-500 81-433m, 81-F-517 81-438m, 81-F-421 81-439m, 81-F-495 82-440m, 81-F-496	32.6 16.9 9.8 ND 3.6, 3.3 10.2 ND ND ND ND
Sun Oil, Missile AV-10, 81-464 Sun Oil, Missile FTM, 14R1, 81-463M Batch 67, A/V, 443-7859 Batch 68, A/V, 443-7859 Batch 69, A/V, 443-7859 81-498-M, 81-F-588	ND ND 14.6 12.4 6.5 0.5

aTPP - triphenylphosphine.

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10. VISIBLE LIGHT ABSORBANCE FOR JP-9 SOLUTIONS OF OIL BLUE A DYE

Two solutions of JP-9 fuel were prepared containing 1.0 and 11.0 milligrams per liter of Oil Blue A dye. Absorbance in the visible spectral region was then measured for each solution over the wavelength rage of 360 to 800 nanometers. The measurements were made on duplicate samples of each solution using a Cary Model 219 Spectrophotometer containing 1.000 centimeter sample cells and undyed JP-9 fuel as the reference fluid. Absorption spectra for the second replicate of each dye solution are presented in

bND - not detected, less than 0.3 ppm.

Figure 253. Absorption maxima occured at wavelengths 597 and 647 (strongest) nanometers, and absorbance values at these wavelengths for the duplicate samples of each dye solution are shown in Table 211.

TABLE 211. ABSORBANCE VALUES FOR JP-9 FUEL SOLUTIONS OF OIL BLUE A DYE

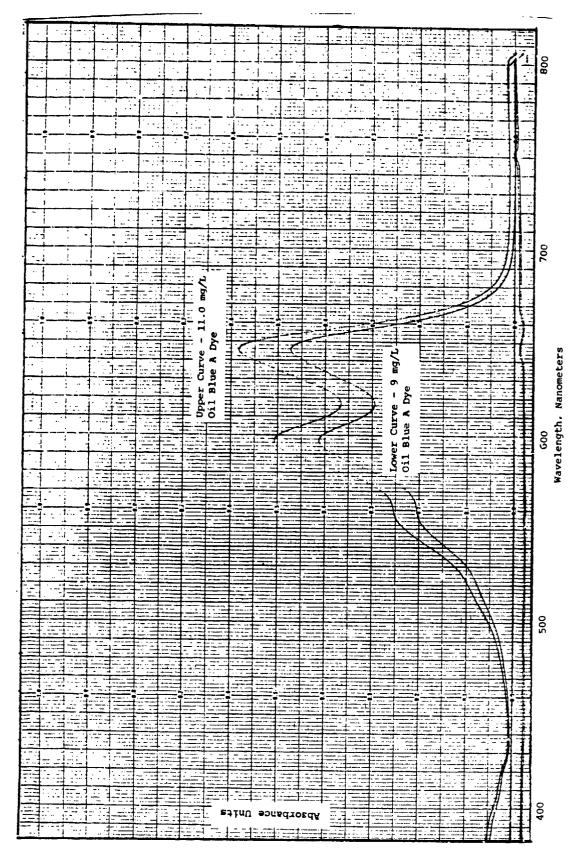
Dye Concentration:	11.0 mg/Liter		9.0 mg/Liter	
Sample Replicate:	1	2	1	2
Absorbance at 646 nm:	0.580	0.582	0.485	0.474
Absorbance at 597 nm:	0.504	0.506	0.424	0.412

11. INVESTIGATION OF DYE PRECIPITATION FROM JP-9 FUEL AT -54°C

JP-9 fuel was prepared with three concentrations of Oil Blue A dye: 5 mg/liter, 10 mg/liter, and 20 mg/liter. After verifying that all dye was in solution, quart bottles of each dye solution were stored at -54°C for a period of 3 days. A visual observation of the bottles while still at -54°C was then made to determine if any of the dye had precipitated. No precipitate was detected. The dye solutions were then forwarded to AFWAL/POSF for long-term storage evaluation.

12. INVESTIGATION OF LOW TEMPERATURE TURBIDITY IN JP-10

This investigation was conducted to establish the cause of turbidity observed in JP-10 as it is cooled to a temperature of -65°F. The haze first appears at approximately -20°F. It was felt that if the turbidity is caused by the release and subsequent entrapment of dissolved air, a significant fuel system problem may be indicated. Fuel system icing inhibitor (FSII), ethylene glycol monomethylether, is used at the 0.15% level in JP-10. A preliminary experiment by the Air Force initiator of this work demonstrated that FSII has a limited solubility in JP-10 at



igure 253. Absorption spectra of JP-9 dye solutions.

red temperatures. This material was thus suspected to be the cause of the turbidity.

Test Procedure

The first experiment was designed to determine the relative effects of dissolved air and FSII on low-temperature turbidity. Two two-liter specimens of JP-10 were prepared, one containing FSII at the specification level and the other containing no FSII. Both specimens were heated to 140°F and thoroughly saturated with air while at that temperature. A fritted glass gas dispersion system was used for that purpose. The samples were then sealed in separate glass containers each having 3.0% ullage. Both containers were cooled to -65°F and observed for cloudiness. sample containing FSII was found to become very milky, while the one without FSII was less turbid but still quite hazy. bubbles of approximately the size found in a carbonated beverage were observed to cling to the inner wall of each vessel, or to rise to the surface of the fuel. The specimens were withdrawn from the cooling bath and allowed to warm to room temperature. The cloudiness almost completely disappeared after 30 minutes, whereupon the unopened containers were again cooled. Exactly the same effect as before was observed except that fewer rising bubbles were noted this time.

The conclusions drawn from this experiment were that while FSII caused cloudiness at lower temperatures, another factor also contributes to the turbidity.

A second experiment was conducted to determine whether the low-temperature cloudiness in the JP-10 was due to trace amounts of dissolved water. The FSII-free JP-10 was processed through a drying column containing anhydrous sodium sulfate followed by a mixture of chromosorb-P/phosphorus pentoxide (P_2O_5) . Upon cooling the dried fuel and a similar undried specimen, the cloudiness was

observed in both samples with no detectable difference in intensity. From this experiment, it was concluded that water is not a cause of haziness.

A third experiment involved comparison of two dried, FSII-free fuels, one being saturated with air at 140°F while the other was completely degassed by subjecting it to a vacuum near its vapor pressure, for a period of one hour. Cooling of these specimens resulted in both having the same degree of turbidity. The vessel containing the air-saturated specimen exhibited the macro-bubbles rising to the surface, as described above.

Summary and Conclusions

Fuel system icing inhibitor at the specification level of 0.15% was shown to be insoluble in JP-10 at -65°F. It is thus a major cause of low-temperature turbidity in this fuel. Haziness, how-ever, appears to a smaller extent in FSII-free JP-10 and it has been shown to be unrelated to water content or to dissolved gases. It can be concluded that the turbidity is likely an endogenous phenomenon of the JP-10 itself, probably being due to a minor fuel component having limited solubility in JP-10 at temperatures approaching -65°F.

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APPENDIX

SPECIFIC TEST METHODS FOR FUEL CHARACTERIZATIONS DESCRIBED IN THIS REPORT

Many physical and chemical property tests were repeatedly conducted for a number of the projects described in this report. For the sake of conciseness and to avoid repetition, the test methods are described in this Appendix only.

DENSITY AND SPECIFIC GRAVITY

This method covers the laboratory determination, using a pyrex dilatometer, of the density of fuels normally handled as liquids. The dilatometer method is most suitable for determining the density of mobile transparent liquids ranging from 100°F to -65°F.

Summary of Method - The liquid is introduced into a clean, weighed dilatometer which is then reweighed. The sample is brought to the prescribed temperature by immersing the dilatometer in a vertical position into a constant temperature bath. After temperature equilibrium has been reached, the dilatometer scale is read by means of a cathetometer.

<u>Definition</u> - Density = the mass of liquid per unit volume at prescribed temperature. In this method, the unit of mass is the gram and the unit of volume, the milliliter.

Apparatus

- (1) A calibrated dilatometer constructed of pyrex glass.
- (2) Thermometers conforming to specifications of American Society for Testing and Materials.
- (3) Constant temperature baths. Water and ethanol are suitable media for temperatures ranging from 100°F down to -65°F.
- (4) Cathetometer
- (5) Dilatometer holder
- (6) Stopper for dilatometer
- (7) Support to hold the dilatometer in approximately a vertical position while weighing, such as a small beaker.
- (8) Analytical balance
- (9) Hypodermic syringes and needles
- (10) Laboratory detergent preparation for glassware

- (11) Toluene, reagent grade
- (12) Distilled water
- (13) Reagent grade acetone

Preparation of Apparatus - Thoroughly clean the dilatometer with chromic acid cleaning solution, rinse well with distilled water and dry at 105 to 110°C, or rinse the dilatometer with pure dry acetone and dry by applying an aspirator to the opened end of the dilatometer. Cleaning should be performed in this manner in order to have a sharply defined miniscus during calibration of the dilatometer. Ordinarily, the dilatometer may be cleaned between test determinations by washing with a suitable solvent, such as toluene, and rinsing with pure dry acetone. Periodic cleaning with glassware detergent solution is recommended.

Calibration of Apparatus - Determine the volume held by the dilatometer when equilibrated at various test temperatures by means of a cathetometer. This is called the K value for that temperature. Freshly-boiled and cooled distilled water can be used for calibrating at 70°F and 100°F and good reagent grade solvents can be used for determining the K values at lower temperatures. Densities of the solvents at low temperature can be determined by using the International Critical Tables and the density of water at various temperatures can be found in the Handbook of Chemistry and Physics. When using the cathetometer, measure the span between two graduation marks on the dilatometer. This will be approximately 0.190 cm for a graduation interval of 0.05 ml. This should be measured at several places to assure consistency. Having determined the span of a 0.05 ml interval, the volume of the liquid can then be interpolated. precise to determine the volume by the cathetometer than to estimate it by visual observation.

<u>Procedure</u> - Adjust a constant temperature bath to maintain the prescribed temperature. weigh the clean, dry dilatometer and stopper to the nearest 0.1 mg and record the weight.

Fill the dilatometer to approximately the 1.5 ml graduation mark on the dilatometer by means of a hypodermic syringe. Remove any bubbles that might have been formed while transferring the sample.

Weigh the stoppered dilatometer and sample to the nearest 0.1 mg. Record the weight. Place the dilatometer in a suitable holder in the constant temperature bath adjusted to the test temperature within ±0.05°F. When the sample has reached equilibrium (about 15 minutes) take readings of the miniscus by means of a cathetometer. Read the cathetometer to the nearest 0.005 cm.

Take several readings of the miniscus until reproducible readings are obtained. After consistent readings have been made, remove the dilatometer from the bath and clean with a suitable solvent, rinse with pure dry acetone and proceed to the next test.

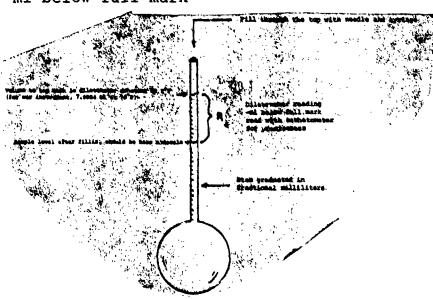
Calculations

density,
$$g/cc = \frac{m}{K-R}$$

where m = mass of sample in grams

K = dilatometer constant at given temperature, volume of dilatometer to full mark

R = dilatometer reading at given temperature, ml below full mark



SIMULATED DISTILLATION BY GAS CHROMATOGRAPHY

Chromatographic-simulated distillations were conducted on hydro-carbon fuels using ASTM Method D 2887. Fuel components were eluted in boiling point order, and actual boiling points were assigned to retention time data by correlation with a calibration curve obtained from a standard hydrocarbon mixture. Data acquisition was accomplished with a Hewlett-Packard 3356 laboratory data system A program had been designed to record area slices of the chromatogram every ten seconds during the chromatographic analysis, and to provide cumulative area slice data as well as a running percent of the total chromatogram area at any given tensecond interval.

HYDROCARBON TYPES BY MASS SPECTROMETRY

Hydrocarbon type analyses were conducted by three separate mass spectral methods, depending on the fuel. A modification of ASTM Method D 2789 was used for JP-4 and gasoline-type fuels. ASTM Method D 2425, which first requires an ASTM D 2549 separation of the fuel into aromatic and paraffinic fractions, was utilized mainly for diesel fuels. Monsanto Method 21-PQ-38-63, developed for hydrocarbon feed stocks with an average carbon number in the range of 12 to 13, was used for JP-8 type fuels. Nonstandard fuels were sometimes analyzed by more than one mass spectral method. All of these analyses are based on the summation of characteristic mass spectral lines for each compound type. A matrix of n equations, relating each of n hydrocarbon types to the summed peak values, is constructed. A computer solution of these simultaneous equations provides a quantitative measure of each compound type present.

As noted above, ASTM D 2425 analysis must be preceded by a separation of fuel aromatics from nonaromatics using a procedure such as that described in ASTM D 2429. The D 2549 method, as currently

presented in Part 24 of the 1980 Annual Book of Standards, required a small procedural modification in order to be used for JP-8. This modification did not change the essential features of the separation, but only involved the method for removing the chromatographic solvent. The modified methodology was developed in MRC laboratories and has been employed for a number of years. An official modification of ASTM D 2549 to achieve the same effect is under study by ASTM Committee D-2 on Petroleum Products and Lubricants. By the ASTM D 2549 procedure, a steam bath is employed to evaporate solvent from the fractions obtained by elution chromatography. In the MRC modification, no heat is applied. Instead, a stream of dry nitrogen is used for desolvation. Evaporation of solvent, in fact, reduces the temperature to below ambient. After the major part of the solvent has evaporated, the weight of the fraction is carefully monitored as the final traces of solvent are removed. Complete removal of solvent is signalled by a marked decrease in the slope of the time/weight loss curve, or in some cases, by the attainment of a constant weight. analysts are experienced in this procedure, which requires a short period of dedicated attention for the processing of each fraction by the analyst.

GROSS AND NET HEAT OF COMBUSTION

Heat of combustion of fuels was determined with an isothermal calorimeter according to ASTM D 240-76. A weighed sample was burned in an oxygen bomb calorimeter under controlled conditions. The heat of combustion was computed from temperature measurements before, during, and after combustion, with allowance for thermochemical and heat transfer corrections. Data were reported as Btu/lb (gravimetric).

The net heat of combustion of a fuel is the amount of heat released when liquid fuel is burned to yield gas-phase water and carlon dioxide. It is a lower value than the gross heat of

combustion which assumes liquid phase water to be the product. The difference between the net and the gross values is equivalent to the latent heat of evaporation of the water formed on burning, which depends on the amount of hydrogen in the fuel. For net heat of combustion calculation, the hydrogen content of each fuel was measured at AFWAL/POSF for use in the following relationship.

$$H_n = H_g - 91.23 \times H$$

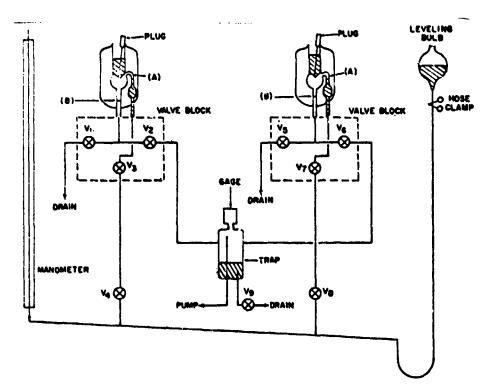
where H_n = net heat of combustion, Btu/lb H_g = gross heat of combustion Btu/lb H = weight percent of hydrogen in the sample.

TRUE VAPOR PRESSURE

True vapor pressure is the maximum vapor pressure that a volatile mixture such as an aircraft fuel can exert at a given temperature. In theory, this property should be measured in the absence of sample vapor because vaporization of a portion of the sample changes the composition of the mixture and thus changes the vapor pressure. In practice, true vapor pressure must be measured at such a small vapor-to-liquid ratio that any change in the composition of the fluid produces a change in vapor pressure which is negligible, i.e., within the experimental error of the method.

For true vapor pressure measurements as a function of temperature, a micro vapor pressure apparatus as described in ASTM D 2551-80 was used. The apparatus incorporates a mercury-sealed orifice for sample introduction, and the entire unit is surrounded by a glass outer jacket through which fluid from a constant temperature bath is circulated. Using this device, a known volume of sample is introduced into an evacuated, temperature-controlled chamber of known volume.

A schematic diagram of the micromethod apparatus is shown below as it appears in the 1981 Annual Book of ASTM Standards, D 2551-80. A fuel sample is introduced through the mercury reservoir at the top of the bulb by means of a mercury displacement pipette.



The sample enters the chamber below which has a volume of 5 ml ±0.03 ml between fiducial marks A and B. After introduction of the sample, the mercury is adjusted to the fiducial marks by means of the leveling bulb and/or drain. Vapor pressure is recorded from a mercury manometer attached to the device. The pressure measured, in actuality, is due to the sample vapor pressure and the partial pressure of any dissolved air.

The object of ASTM Method D 2551 is the determination of vapor pressure values that are comparable to those obtained by the Reid vapor pressure technique. In measurements within this laboratory, the apparatus has been employed for true vapor press re determinations which, of course, must not include the effects of dissolved gases (atmospheric constituents). The measurements are, thus, preceded by an operation to cryogenically degas the sample, thereby

removing permanent gases while retaining even the most volatile hydrocarbons. In the cryogenic degassing procedure approximately 50 ml of fuel is placed in a glass vessel that is connected to a vacuum system through a wide-bore, greaseless stopcock. The sample vessel is submerged in liquid nitrogen until the fuel sample is frozen solid. While the fuel is still solid, the stopcock is opened and the vessel is evacuated to approximately 5 µm pressure. The stopcock is then closed and the fuel is allowed to melt. As the fuel becomes liquid, bubbles of dissolved gas are released.

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The fuel is then frozen a second time and the vessel is again evacuated. By melting the fuel in the isolated vacuum vessel again, air is released but not nearly the quantity obtained the first time. This freeze/melt cycle is repeated until no increase in pressure is noted in the vessel after the fuel has been melted and refrozen. By strict adherence to this procedure, no opportunity exists for loss of sample vapors. The fuel is cooled to -70°F to recondense vapors before removal for vapor pressure testing.

The importance of removing dissolved air from the fuel can be understood by considering the solubility of air in JP-4. The Ostwald solubility coefficient (volume of dissolved gas per unit volume of liquid fuel, measured at the temperature and pressure of saturation) for air in JP-4 is approximately 0.19^a at 75°F. Thus the 2 ml volume of JP-4 used for vapor pressure measurement can contain 0.38 ml of air. The volume of the chamber in the apparatus which is occupied by vapor is 3 ml. Sample is introduced into the vacuum of the apparatus through a mercury-pool seal. Some outgassing at this time is almost assured. Consideration of simple gas laws shows that if the entire volume of dissolved air is released, 0.38 ml at atmospheric pressure, pressures

^aF. N. Hodgson and A. M. Kemmer, AFAPL-TR-76-26, March 1976.

up to 90 mm of mercury could result from the dissolved air. It is, thus, imperative that the bulk of the dissolved air be removed for true vapor pressure measurements.

The volume of the micro apparatus is 5 ml. By introduction of 1 ml of fuel, a vapor-to-liquid (V/L ratio) of 4:1 is obtained; 2 ml of fuel gives a V/L ratio of 3:2. Tests must be conducted at several V/L ratios to determine the point at which vapor pressure does not measurably increase with decrease in the vapor volume. Usually 2 ml of sample (V/L = 1.5) was sufficient to obtain this condition.

Classically the vapor pressure temperature relationship is expressed by:

$$log P = A - B/T$$

where A and B are constants, P = absolute pressure and T = absolute temperature. The line resulting from a plot of this equation on semilog paper is useful over a limited range for estimating vapor pressure at temperatures intermediate to, or just beyond, those at which measurements were taken.

The repeatability and reproducibility figures provided in D 2251 should be valid for the true vapor pressure procedure. ASTM D 2551 cites the repeatability of the method (maximum difference between successive determinations by the same operator) to be 4 mm at the 95% confidence level. Reproducibility (for different operators in different laboratories) is cited as 15 mm at the 95% confidence level. In practice, differences between replicate vapor pressure measurements in this laboratory seldom exceed 2 mm and are frequently within 1 mm.

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s recommended that measurements at -20°F generally should not be conducted, but rather these values should be obtained by extrapolation of the log P versus $\frac{1}{T}$ plot which usually exhibits good linearity. In general it is recommended that vapor pressures below approximately 5 mm should not be measured directly as they are more reliably obtained by extrapolation.

SURFACE TENSION AS A FUNCTION OF TEMPERATURE

Surface tension was measured as a function of temperature using the capillary-rise method. Surface tension in dynes cm⁻¹ is given by the expression:

$$\tau = \frac{\text{rhdg}}{2\cos \theta}$$

where $d = density of the liquid, <math>g/cm^3$

h = height of the column of liquid, cm

 $g = acceleration of gravity, cm/s^2$

r = radius of the capillary, cm

 θ = contact angle, degrees

For most materials the contact angle (θ) was essentially zero and $\cos \theta$ was equal to one. However, when the material being measured wetted the capillary walls poorly, as in the case of some high density fuels, the contact angle was measured. A Gaertner contact angle goniometer apparatus was used for this measurement. From the data generated, a linear temperature/surface tension relationship is established.

KINEMATIC VISCOSITY

Kinematic viscosity was determined as a function of the temperature for all fuels and fuel components using the procedure and equipment described in ASTM Method D 445-79. Viscosity data were plotted on standard ASTM viscosity-temperature charts which are useful for the estimation of kinematic viscosities at temperatures other than those at which measurements were conducted.

AIR AND NITROGEN SOLUBILITY AS A FUNCTION OF TEMPERATURE

The amount of air or nitrogen soluble in fuels at saturation was measured using a special degassing chamber and cold trap on the inlet of a CEC 21-103C mass spectrometer. The vacuum of the mass spectrometer inlet was used to aid in degassing the specimens. The degassing vessel was arranged so that constant agitation of the sample was possible. For viscous samples, heating was also used in the degassing process. Air and hydrocarbon vapors removed from the sample are passed through a cold trap where most of the hydrocarbons remain. The amount of air was determined in the desorbed gases as the total of the nitrogen, oxygen, argon, and carbon dioxide. For nitrogen solubility measurements, only the value for desorbed nitrogen was used.

Instrument calibration was achieved by introducing known volumes of air or nitrogen into the system. The determined value was independent of the amount of hydrocarbons in the vapor. Fuels are saturated with air in a distillation flask to which a reflux condenser was attached. The flask was placed in a constant temperature bath maintained at the desired temperature. A fritted-glass gas dispersion tube was immersed into the fuel through which CGA Grade E compressed breathing air or prepurified nitrogen was passed. Water from an ice bath was circulated through the jacket of the condenser by means of a small pump. This procedure prevents loss of volatile components during saturation of the fuel.

Solubility is expressed as an Ostwald coefficient, defined as the volume of air per unit volume of fuel, both volumes measured at the temperature of saturation.

SPECIFIC HEAT AS A FUNCTION OF TEMPERATURE

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The specific heat of solids and liquids was measured with a Perkin-Elmer differential scanning calorimeter, Model DSC-1.

The calorimeter measures the rate of heat flow into a sample (which is proportional to specific heat) whose temperature is being programmed at a linear rate. The specific heat is then calculated by comparing the rate of heat flow for the weighed sample to the rate of heat flow of a weighed standard (synthetic sapphire) for which the change of specific heat with temperature is accurately known.

The specific heat can be measured anywhere between -100 and +500°C with milligram quantities of sample contained in standard cups or hermetically-sealed cups. The ultimate precision of the method is 0.3% or better, which approaches the precision of adiabatic calorimetry.

THERMAL CONDUCTIVITY AS A FUNCTION OF TEMPERATURE

Thermal conductivity was determined using a Monsanto-built transient hot wire apparatus. In this procedure a constant heating current is applied abruptly to a resistance wire immersed in the fuel. The change in temperature of the wire following application of the current is obtained from the observed change of voltage across the wire and the known resistance-temperature characteristics.

This method has been established as being among the best available for routine thermal conductivity measurements. Complete instrumentation for this measurement is located at our St. Louis laboratories where all work of this nature was conducted. Personnel from that laboratory have published the results of a detailed study on the measurement of the thermal conductivity of liquids.

b"Rapid Measurement of Liquid Thermal Conductivity by the Transiient Hot-Wire Method," W. N. Trump, H. W. Luebke, L. Fowler, and E. M. Emery, Rev. Sci. Instrum. 48, 47, 1977.

DIELECTRIC CONSTANT AS A FUNCTION OF TEMPERATURE

Dielectric constants were routinely measured as a function of temperature at 400 Hz for all fuels and fuel components. Measurements were made in a three terminal guarded cell relative to air at the same temperature. A General Radio 1615A capacitance bridge and guard circuit were used in the determination. Linear regression analyses were performed on the data to determine the dielectric constant as a linear function of temperature.

GAS CHROMATOGRAPHIC ANALYSES

Gas chromatographic analyses conducted on high density fuels employed the same instrument and similar analytical conditions as used for jet fuels.

A Perkin-Elmer Model 3920B gas chromatograph having a 50-meter by 0.01-inch glass open tubular column coated with SF-96 stationary phase was used as required. Other parameters were:

Detect : type - flame ionization

Carrier gas - helium

Flow rate - 3 ml/min

Typical temperatures program - column initially 60°C, held for 4 min

Program rate - 8°C/min

Final temperature - 190°C

Injection port - 300°C

Data were recorded and processed using a Hewlett-Packard 3356 laboratory data system in most cases.

WATER SOLUBILITY

Water solubility, the quantity of water which will dissolve in a fuel, was determined as a function of temperature during the

course of this work. For this measurement, the fuel is saturated with water by placing a vessel containing the fuel sample, along with an excess of distilled water, into a constant temperature both. The fuel is then stirred with a small mixer for approximately 18 minutes. The water is then allowed to separate from fuel so that no turbidity remains. Samples of the fuel are then removed for analysis.

A coulometric-type Karl Fischer titrimeter was used for the determination of water. In this system, reagent is generated by an electrical current, thus no standardization is required. The end point is electronically detected. For best precision about 1,000 micrograms of water are required in the specimen. Thus to attain a 10 ppm detection limit, a sample weight of 100 grams was used.

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